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Preparation and Tribological Behavior of γ-AlOOH/ Hexagonal Boron Nitride Composites as Additives in Water-Based Lubricant

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Recently, water-based lubricant has received extensive attention with the enhancement of environmental awareness and economic considerations. A series of novel γ -AlOOH/h-BN composites were successfully prepared by simple hydrothermal method and employed as additive to improve tribological behaviors of water-based lubricant. The morphologies of γ -AlOOH nanoparticles were influenced in different alkaline environments, which could be attributed to the formation of Al-triethylamine (Al-TEA). Furthermore, when the quantity of BN is same, the granular γ -AlOOH nanoparticles under TEA condition owing to a smaller size presented the better anti-friction property. The dispersibility and stability of BN in water were improved via loading AlOOH nanoparticles onto BN. Compared with pure BN, the nanocomposites can be stable in water for 24 h. The tribological behaviors were studied by four-ball tribological machine, the results showed that obtained samples exhibited outstanding anti-friction and anti-wear properties both in pure water and in water-glycol (mass ratio is 55%:45%) lubricant.

Keywords	γ-AlOOH/h-BN, anti-friction, anti-wear, composites,
	water-based lubricant

1. Introduction

Friction and wear have caused various problems such as unnecessary energy wastage, environmental pollution, equipment damage and so on (Ref 1-4). Therefore, controlling friction and wear are treated as a crucial strategy for protecting the environment and improving energy efficiency (Ref 5, 6). Recently, many materials like lubricants were designed and used to reduce the friction and wear (Ref 7-11). Thereinto, synthetic oil and mineral oils are difficult to degrade in nature as oil lubricant (Ref 12). Compared with the oil lubricants, water-based lubricants have many unique advantages like high compatibility with the environment, low cost and good environmental compatibility (Ref 13, 14). However, waterbased lubricant has poor tribological properties, which can be effectively improved by introducing functional additives to water-based lubricant. Lubricant additives improve the bearing capacity and oxidation resistance, reduce friction and reduce wear (Ref 15). Xiong et al. (Ref 16) synthesized N-containing heterocyclic imidazoline compounds which improved tribological performances of the water-glycol.

Two-dimensional (2D)-layered materials incorporating graphene, transition metal dichalcogenides (TMDs), MXene (Ti₃C₂) and hexagonal boron nitride (h-BN) have drawn enormous attention because of the excellent tribological behaviors (Ref 17-19). Additionally, BN has become a promising candidate in tribological filed owing to its splendid thermal conductivity performance, chemical stability, electrical insulation property and low friction coefficient. BN mainly consisted of B and N atoms which are connected via strong covalent bond. The layers of h-BN are connected by weak Van der Waals forces, making it easier to shear along base surface of wafer layer structure under sliding pressure (Ref 20, 21). During the process of slide, h-BN can enter the contact rubbing areas and then avoid the direct contact of friction pairs, friction and wear are effectively reduced (Ref 22-24). However, BN is poorly dispersed in water-based lubricants, which remains a challenge (Ref 25).

Many inorganic nanoparticles possess small particle size so that they can fill with micro-roughness (Ref 26). In the meantime, nanoparticles may precipitate and form protective film ascribed to the high surface energy, they also owe the effect of repairing or self-repairing on the worn surfaces (Ref 27). Metal oxide nanoparticles have been proved to have good anti-friction effect. Wang et al. (Ref 28) successfully synthesized Fe₃O₄ with different morphologies (nanospheres, nanowires and nanosheets), which can effectively improve its tribological properties when used as an additive to liquid paraffin. Zhao et al. (Ref 29) used the hydrothermal method to prepare water-soluble CuO nanoparticles with different morphologies (nanobelt, nanorod and spindle). As a water-based lubricant additive, CuO nanoparticles can enter the friction surface to form a friction protective film, which can prevent the direct contact of the friction pair. In addition, dispersing the nanoparticles onto the 2D-layered material can improve the agglomeration of the nanoparticles. At the same time, the dispersibility of 2D-layered materials was improved in the lubricant (Ref 30).

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Herein, we successfully prepared a series of novel γ -AlOOH/h-BN composites employed the simple hydrothermal methods to load the γ -AlOOH nanoparticles onto the h-BN. The structures and surface morphologies of γ -AlOOH/h-BN samples were verified by FTIR, XRD, SEM and TEM. Then, the tribological properties of γ -AlOOH/h-BN samples were studied as additives in water and water-glycol, respectively. In consequence, this paper provided a feasible strategy for preparing water-based lubricant additives.

2. Experimental Sections

2.1 Materials and Reagents

Hexagonal boron nitride (h-BN) was bought from Tianyuan Chemical Research Institute. Potassium hydroxide (KOH), aluminum nitrate hydrate (Al(NO₃)₃·9H₂O), triethylamine (TEA) and sodium citrate were gained from Sinopharm Chemical Reagent Co. Ltd. Cetyltrimethyl ammonium bromide (CTAB) was obtained from Beijing Aoboxing Biotechnology Co. Ltd. All chemicals were analytic grade and for direct use.

2.2 Preparation of y-AlOOH/h-BN

The sodium citrate and CTAB (molar ratio 1:2) were dissolved into 70 mL deionized water and then stirred for 20 min. Then, 3.5 g of Al(NO₃)₃·9H₂O was added to the solution and stirred until it completely dissolved. The 0.18 g, 0.26 g, 0.35 g, 0.48 g and 0.7 g of h-BN powders were dispersed to the above solution and ultrasonicated for 3 h; then, dispersed solution A was obtained. Finally, the KOH and TEA were used to provide alkaline environment, respectively.

The 2.35 g of KOH was dissolved in 5 mL deionized water and added to the dispersed solution A. After stirring at 50 °C for 45 min, the mixtures were transferred to the 100 mL PTFE reactors and heated at 180 °C for 15 h. After the reaction finished, the products were washed with deionized water and ethanol. The γ -AlOOH/h-BN (1-5) samples were obtained after drying at 60 °C for 12 h in the oven. The pure γ -AlOOH (1) nanoparticles were prepared under the same condition without BN. The reactions in hydrothermal process are as follows:

$$Al^{3+} + 3OH^{-} \rightarrow Al(OH)_{3}$$
 (Eq 1)

$$Al(OH)_3 \rightarrow AlOOH + H_2O$$
 (Eq 2)

The appropriate amount of TEA was slowly added to the dispersed solution A until the pH reached 11. After stirring at room temperature for 30 min, the mixture was transferred to the 100 mL PTFE reactors and heated at 190 °C for 20 h. After the reaction finished, the products were washed with deionized water and ethanol. The γ -AlOOH/h-BN (6-10) samples were obtained after drying at 60 °C for 12 h in an oven. The pure AlOOH (2) nanoparticles were gained using the method. The reactions in hydrothermal process are as follows:

$$Al^{3+} + TEA \rightarrow [Al - TEA]^{3+}$$
 (Eq 3)

$$[AI - TEA]^{3+} + 3OH^{-} \rightarrow Al(OH)_{3} + TEA \qquad (Eq 4)$$

2.3 Characterization

The chemical bonds of pure h-BN, γ -AlOOH and γ -AlOOH/ h-BN composites were investigated by Fourier transform infrared spectra (FTIR, TENSOR-27, Perkin Elmer, USA); the KBr was used as the matrix (4000-400 cm⁻¹). Phase and crystallization of prepared composite materials were surveyed by X-ray diffraction analysis (XRD, AXIS D8, Bruke, Germany). Scanning electron microscopy (SEM, SU8000, JEOL, Japan) was employed to investigate the surface morphology of composites. Transmission electron microscopy (TEM, JEM-2100, JEOL, Japan) was used to further confirm the size and morphologies of the composites.

2.4 Tribological Test

The tribological behaviors of water and water-glycol (mass ratio 55%:45%) with y-AlOOH/h-BN (1-10) were characterized via tribological test. Water-based lubricants with y-AlOOH/h-BN (1-10) additives were prepared at concentration 0.03, 0.06, 0.09, 0.12 and 0.15 wt.%. The average COF was obtained by using a four-ball friction tester (MMW-1A, Jinan Yihua, China). The friction pair was consisted by an upper ball, three lower balls ($\Phi = 12.7$ mm, GCr15). Before the test started, the steel balls were cleaned by acetone ultrasonic for 30 min to remove surface contamination. Then, 8 mL of lubricant was added until the friction surfaces could be covered. The tribological performance tests were proceed at room temperature with 100 N load and test duration of 30 min; rotation speed was set to 120 r/min. The morphologies and WSD of three bottom balls were observed by YW MS2300D optical microscope with ± 0.01 mm accuracy. All tribological tests were repeated three times to obtain mean value under the same test conditions.

3. Results and Discussion

3.1 FTIR Analysis

The FTIR spectra of h-BN and y-AlOOH nanoparticles and γ-AlOOH/h-BN (1-10) are shown in Fig. 1. The characteristic peaks at 3300 and 3086 cm⁻¹ belong to symmetric and asymmetric stretching vibrations of Al-OH. The shoulder peak of 1165 cm⁻¹ is related to symmetric and asymmetric bending vibrations of Al-OH. The tensile vibration absorption peak of Al-O-Al was located at 1070 cm^{-1} . The peaks at 742, 619 and 492 cm⁻¹ were associated to the vibration of AlO₆ octahedron modes, which corresponded to torsional vibrations, stretching vibration and bending stretching vibration of Al-O, respectively (Ref 31-33). The 1632 cm^{-1} belongs to bending vibrations of O-H (surface adsorbed H₂O molecule) (Ref 34). The peak of 1369 and 816 cm⁻¹ was ascribed to the in-plane stretching vibration absorption peak of BN and out-of-plane bending vibration absorption peak of BN (Ref 35, 36), respectively. As the BN addition increased, the characteristic peaks intensity of BN gradually increased, while peak intensity of y-AlOOH



Fig. 1 FTIR spectra of (a) h-BN, γ -AlOOH (1), γ -AlOOH/h-BN (1-5) and (b) h-BN, γ -AlOOH (2), γ -AlOOH/h-BN (6-10)



Fig. 2 XRD patterns of (a) h-BN, γ -AlOOH (1), γ -AlOOH/h-BN (1-5) and (b) h-BN, γ -AlOOH (2), γ -AlOOH/h-BN (6-10)

gradually decreased, which indicated that the loading of γ -AlOOH particles on the surface of h-BN would be influenced by quantity of BN.

3.2 XRD Analysis

Figure 2 exhibited XRD patterns of h-BN, γ -AlOOH, γ -AlOOH/h-BN (1-10) samples. As the figure shown, the peaks at $2\theta = 14.28^{\circ}$, 28.15° , 38.55° , 49.19° , 55.17° and 64.9° could be observed, which were corresponded to the crystal plane diffraction peaks of (020), (120), (031), (220), (151) and (002) of orthorhombic structure γ -AlOOH (JCPDS card no. 021-1307), respectively (Ref 34, 37). The characteristic diffraction peaks at $2\theta = 26.79^{\circ}$, 41.69° , 43.84° corresponded to (002), (100), (101) three diffraction crystal planes of h-BN (JCPDS No. 34-0421), respectively (Ref 36, 38, 39). XRD results are consistent with FTIR spectra, indicating that γ -AlOOH is successfully loaded onto h-BN.

3.3 Morphology Analysis of γ -AlOOH/h-BN (1-10)

Figure 3 showed the SEM pictures of the γ -AlOOH nanoparticles and γ -AlOOH/h-BN (1-10) composites. The h-BN demonstrated stacked sheet structure with a diameter of about 8-10 μ m, while γ -AlOOH particles under different alkaline conditions all presented nanoscale size. There are

obvious differences in morphology, γ -AlOOH (1) nanoparticles exhibited thin nanosheet structure and were prone to agglomerate randomly in Fig. 3(a), but γ -AlOOH (2) nanoparticles showed granular morphology of agglomeration (Fig. 3g). The surface of γ -AlOOH/h-BN (1-10) samples became obviously rough than pure BN sheet, indicating that the γ -AlOOH nanoparticles were successfully loaded on the BN sheet. Indeed, the γ -AlOOH nanoparticles were more uniformly distributed and relatively more dispersed on the BN sheet in contrast to pure AlOOH. Meantime, the size and morphology of AlOOH nanoparticles did not change significantly even loading on BN. Additive amount of BN only has an effect of loading capacity of the γ -AlOOH nanoparticles on BN sheet.

For γ -AlOOH/h-BN (1-5) composites, distribution of γ -AlOOH was gradually altered from multi-layer packing to single layer as the amount of BN increased. The γ -AlOOH nanoparticles achieved the optimum dispersion in AlOOH/h-BN (4) sample (Fig. 3e). As the amount of BN further increased, the quantity of AlOOH (1) nanoparticles loading on BN decreases. It could be attributed that the number of nanoparticles is not sufficient to support their formation of single-layer distribution over more BN sheets. The distributed condition of γ -AlOOH nanoparticles on BN in γ -AlOOH/h-BN (6-10) samples could be observed (Fig. 3g-l). Similarly, γ -AlOOH nanoparticles could be loaded on BN sheet as addition



Fig. 3 SEM images of (a) γ-AlOOH (1), (b-f) γ-AlOOH/h-BN (1-5), (g) γ-AlOOH (2) and (h-l) γ-AlOOH/h-BN (6-10)



Fig. 4 TEM images of (a) γ -AlOOH (1), (b) γ -AlOOH/h-BN (4), (c) γ -AlOOH (2) and (d) γ -AlOOH/h-BN (9)

of BN and evenly dispersed on the surface of BN. The γ -AlOOH (2) particles were in an optimal state of dispersion at the Fig. 3(K). As the amount of h-BN continued to increase, the γ -AlOOH nanoparticles which loaded on BN sheets decrease. SEM pictures confirmed that γ -AlOOH was successfully loaded on BN sheets, and different bases had a great impact on morphology of AlOOH nanoparticles. The addition of BN did

not change the size and morphology of nanoparticles, but dispersion state was affected with the amount of BN addition.

Figure 4 showed TEM images of γ -AlOOH (1), γ -AlOOH (2), γ -AlOOH/h-BN (4) and γ -AlOOH/h-BN (9) samples. As shown in Fig. 4(a), γ -AlOOH (1) nanoparticles were irregular folded sheet with the diameter of about 100 nm. Due to the different orientations of each sheet, the particle clusters

presented a two-dimensional sheet stacking state. After loaded on BN (Fig. 4b), γ -AlOOH (1) nanoparticles were orderly distributed to BN surface with the single layer, which is in accordance with Fig. 3(e). The morphology of γ -AlOOH (2) nanoparticles presented long granular structure with the single particle length of about 20 nm, which shown in Fig. 4(c). Similarly, it can be clearly found in Fig. 4(d) that the particles were evenly distributed on the BN sheet.

3.4 Dispersion and Stability Analysis

Figure 5 showed the dispersion of composites at the concentration of 0.06 wt.% in water-based lubricant. It could be observed that the composites were relatively evenly dispersed in the water-based lubricant at first. After 12 h, all the other γ -AlOOH/h-BN (1-5) samples agglomerated and settled completely except γ -AlOOH/h-BN (3) in the water. The γ -AlOOH/h-BN (6-10) agglomerated and settled completely in the water after 24 h, because of the γ -AlOOH (2) have smaller size in comparison with γ -AlOOH (1). The γ -AlOOH/h-BN (1-5)

10) agglomerated and settled completely in the water-glycol after 24 h. The prepared samples all presented the excellent dispersibility.

3.5 Tribological properties in different lubricants

Figure 6(a-b) displayed the average COFs of γ -AlOOH/h-BN (1-10) composites additives with the concentration of 0.03-0.15 wt.% at 100 N loading. The average COFs of the γ -AlOOH/h-BN (1-10) were all lower than that of pure water, which indicated γ -AlOOH/h-BN (1-10) samples have significant friction-reducing property in water as lubricant additives (shown in Fig. 6). At the same time, it is not difficult to find that the average COFs of the γ -AlOOH/h-BN (1-10) samples decreased first and then increased as the concentration increased. According to Fig. 6(a), when the additive concentration of γ -AlOOH/h-BN (2) sample in water was 0.12%, the average COF value reached minimum which was 0.250. The average COF was reduced by 37.58% than pure water, which showed the best anti-friction effect in γ -AlOOH/h-BN (1-5)



Fig. 5 The optical pictures of (a-b) γ -AlOOH/h-BN (1-10) in water and (c-d) γ -AlOOH/h-BN (1-10) in water-glycol at the concentration of 0.06 wt.%



Fig. 6 Average COFs of (a) γ -AlOOH/h-BN (1-5), (b) γ -AlOOH/h-BN (6-10), average WSDs of (c) γ -AlOOH/h-BN (1-5) and (d) γ -AlOOH/h-BN (6-10) in water

samples. In γ -AlOOH/h-BN (6-10) samples, γ -AlOOH/h-BN (10) exhibited the best anti-friction effect. γ -AlOOH/h-BN (10) had the minimum average COF value of 0.241 at the additive concentration 0.09 wt.% (shown in Fig. 6b), which is 41.20% lower than that without additive in this serious of composites.

The average WSDs of γ -AlOOH/h-BN (1-10) additives are showed in Fig. 6(c-d), which showed a similar trend to the average COF. The average WSDs of γ -AlOOH/h-BN (1-10) were also less than that of pure water, indicating γ -AlOOH/h-BN (1-10) samples have outstanding anti-wear property as lubricant additives. The average WSDs of γ -AlOOH/h-BN (4) at concentration 0.12 wt.% reached the minimum value of 0.382 mm, which was reduced by 30.54% compared with the average WSD in pure water. According to the Fig. 6(d), the average WSDs of γ -AlOOH/h-BN (10) reached the minimum value of 0.374 mm, which was 32.06% lower than the average WSD in pure water, which showed excellent anti-wear effect.

Figure 7 displayed the average COFs of γ -AlOOH/h-BN (1-10) composites containing the concentration of 0.03-0.15 wt.% in the water-glycol (mass ratio 55%:45%) at 100 N loading. It could be known that the average COFs of the γ -AlOOH/h-BN (1-10) were completely less than that of lubricant, which suggested γ -AlOOH/h-BN (1-10) additives show outstanding anti-friction behavior in lubricant. In the meantime, it worth noting that the average COFs of the γ -AlOOH/h-BN (1-10) samples presented the trend of first decreasing and then increasing. When additive concentration of γ -AlOOH/h-BN (1) sample in water-glycol was 0.09 wt.%, the average COF value reached minimum which was 0.0936 (Fig. 7a). Compared with the water-glycol, the average COF was reduced by 55.44%, which showed the best anti-friction effect in γ -AlOOH/h-BN (1-5) samples. As could be seen in Fig. 7(b), γ -AlOOH/h-BN (10) had the minimum COF value of 0.0972 at the additive concentration 0.12 wt.% in the γ -AlOOH/h-BN (6-10) samples. Compared with lubricant, the COF was reduced by 53.72%, exhibiting the best anti-friction effect in γ -AlOOH/h-BN (6-10) samples.

The average WSDs of γ -AlOOH/h-BN (1-10) additives in water-glycol are demonstrated in Fig. 7(c-d) with different concentrations at 100 N loading. Fig. 7(c) showed that the average WSDs of γ -AlOOH/h-BN (1-10) were all smaller than that of water-glycol, exhibiting the outstanding anti-wear behavior of γ -AlOOH/h-BN (1-10) samples as lubricant additives. The average WSDs of γ -AlOOH/h-BN (5) at concentration 0.12 wt.% reached the minimum value of 0.333 mm, which was reduced by 39.45% compared with the average WSDs in pure water-glycol. According to Fig. 7(d), the average WSDs of γ -AlOOH/h-BN (6) reached the minimum value of 0.344 mm, displaying splendid anti-wear performance. Average WSDs was 37.45% lower than the average WSD of water-glycol.

The wear surface morphologies of γ -AlOOH/h-BN (4) and γ -AlOOH/h-BN (10) are exhibited in Fig. 8 at 0.03-0.15 wt.% concentration. The friction surface has many grooves and scratches after pure water lubrication, which was caused by the poor strength of the protective film formed by water on the friction surface (Fig. 8a). When γ -AlOOH/h-BN (4) and γ -AlOOH/h-BN (10) were used as additive of water, the wear scar of the friction surface was significantly relieved in various degrees. The wear scar obviously became shallower, and the



Fig. 7 Average COFs of (a) γ -AlOOH/h-BN (1-5), (b) γ -AlOOH/h-BN (6-10), average WSDs of (c) γ -AlOOH/h-BN (1-5) and (d) γ -AlOOH/h-BN (6-10) in water-glycol



Fig. 8 Wear surface micrograph of (a, g) water, (b-f) γ -AlOOH/h-BN (4) and (h-l) γ -AlOOH/h-BN (10) in water

worn surface became smoother. Meanwhile, the WSD was all obviously reduced, which was consistent with the trend shown in Fig. 6(c-d).

Figure 9 demonstrated the worn surface morphologies of γ -AlOOH/h-BN (5) and γ -AlOOH/h-BN (6) at 0.03-0.15 wt.%

water-glycol concentration. According to Fig. 9(a), the grooves and scratches improved compared with pure water, which was attributed to addition of glycol. After γ -AlOOH/h-BN (**5**) and γ -AlOOH/h-BN (**6**) were added to the lubricant, the wear scar of the friction surface was shallower and the surface became



Fig. 9 Wear surface micrograph of (a, g) water-glycol, (b-f) γ -AlOOH/h-BN (5) and (h-l) γ -AlOOH/ h-BN (6) in water-glycol



Fig. 10 Average COF of γ -AlOOH/h-BN under the same amount of h-BN

smoother. Meanwhile, the variation trend of the WSD is consistent with Fig. 7(c-d).

Figure 10 displayed comparison of average COF values of γ -AlOOH/h-BN composites at the same BN addition amount. According to Fig. 10, the average COF of γ -AlOOH/h-BN prepared under TEA is basically less than γ -AlOOH/h-BN prepared under KOH when addition quantity of BN is same. This may be caused that the BN enters the frictional pairs at the beginning and then penetrates into the grooves which formed the protective films because of the intrinsic lamellar structure (Ref [40-42]). Subsequently, the direct contact of friction surfaces was avoided. The rich -OH groups provided by γ -AlOOH render the composites to better adsorb on the surface of friction, causing the protective film more complete. Furthermore, γ -AlOOH nanoparticles further improve the dispersion and stability of h-BN in water-based lubricant, which makes protective film more stable on the friction pair. The addition of nanoparticles also improved the bearing capacity of 2D

Table 1 Comparison of reduction of average COF for different composites

Materials	Lubricant	Reduction of COF, %	References
Fe ₃ O ₄ /MoS ₂	Water	34.60	(Ref 30)
AlooH/GO	Hydroisomerization base oil (VHVI8)	14.00	(Ref 43)
SiO ₂ /GO	Water-based	20.70	(Ref 46)
AIOOH/BN	Water-based	55.46	Present work

materials, the excellent anti-friction and anti-wear properties of composites may be ascribed synergistic effect of nanoparticles and 2D layer material on the frictional interfaces (Ref [43-45]). Therefore, when BN quantity is same, γ -AlOOH nanoparticles plays a decisive role in friction. γ -AlOOH (2) has smaller size and granular morphology, resulting in the easier access to the friction surface and possessing the smaller average COF.

Table 1 shows the Comparison of Reduction of average COF for different composites. Through comparison, it can be seen that composites synthesized by us showed better anti-friction effect in the water-based lubricant.

4. Conclusion

In conclusion, a series of γ -AlOOH/h-BN samples were successfully synthesized by hydrothermal method. Morphologies of the γ -AlOOH nanoparticles were adjusted by using KOH and TEA to provide different kinds of OH⁻, respectively. The obtained γ -AlOOH/h-BN samples could be used as waterbased lubricant additive because the γ -AlOOH possessed the abundant -OH group. Meantime, all γ -AlOOH/h-BN composites could significantly improve the anti-friction and anti-wear properties of water-based lubricant. The tribological properties depend on the synergistic lubrication between γ -AlOOH and h-BN, the uniform dispersion of γ -AlOOH on the surface of the BN is also beneficial to the formation of a stable friction protective film.

Competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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