3D graphene/hexagonal boron nitride composite nanomaterials synergistically reduce the friction and wear of Steel-DLC contacts

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Abstract
Green tribology is an engineering field, which is dedicated to reducing friction, wear and pollution to the environment. Diamond-like carbon (DLC) films as coating is a very effective green friction material, but its friction of coefficient in the atmospheric environment still needs to be further reduced. Here, a common method for improving the tribological performance of DLC films in ambient atmosphere by adding 3D graphene/hexagonal boron nitride (h-BN) composite nanomaterials as lubricant additive is proposed. By comparing with DLC film and other single additives, we can see that the 3D graphene/h-BN composite nanomaterial as a lubricating additive reduce the friction coefficient and wear rate remarkably. By studying the tribological properties of the surface of the steel ball and DLC film, it is found that the friction protective film containing carbon, nitrogen and boron elements are formed on the surface. The presence of the friction layer can reduce friction efficiently, not only prevent the surface of the steel ball from being excessively worn, but also protect the DLC film from being damaged. The important thing is that it does not contain harmful elements and provides a new reference for the development of the next generation of oil-free lubrication tribology. Accordingly, it is considered an environmentally friendly additive.

KEYWORDS
3D graphene, friction and wear, hexagonal boron nitride, lubricating additive

1 | INTRODUCTION

With the global focus on energy security, environmental sustainability and economic feasibility, researches on low-friction materials, coatings and lubricants have been continuously strengthened in recent years, resulting in the development of many new materials and lubricants.¹,² Lubricants and additives are used to keep friction and wear in optimum condition by physically packing on the surface of the sliding material to avoid direct contact between the two materials.³,⁴ In the past two decades, carbon nanomaterials have attracted great interest due to their excellent...
tribological applications, in particular, two-dimensional materials such as graphene, hexagonal boron nitride and molybdenum disulfide have the lowest coefficient of friction and wear rate, which makes them attractive in terms of improving the efficiency, durability and environmental compatibility of future mechanical systems.\[5,6\] Diamond-like carbon (DLC) film is kind of amorphous carbon, which is composed of sp$^3$ and sp$^2$ bonded carbon atoms.\[7\] It is famous for its high hardness, low friction and excellent wear resistance, which helps to enhance the mechanical and tribological properties of materials. From small mechanical equipment to large automobile industry, as an effective protective coating, DLC film has been widely studied for more than two decades.\[8–10\] Of course, their current greatest application is in the automotive filed because they are now commonly used in injectors, tappets, piston pins and rings, gears, bearings, etc.\[11–13\] The interest in commercializing DLC technology for tribological applications continues to grow.\[14\] Nevertheless, DLC film is greatly affected by the surrounding environment. In a vacuum and dry inert atmosphere, the friction coefficient of hydrogen containing amorphous carbon (a-C:H) film can reach an ultra-low friction state of less than 0.01, while amorphous carbon (a-C) film has a higher friction coefficient (>0.4) in a dry environment. The above-mentioned problems are the key factors affecting the tribological properties of DLC films, and they are also problems that need to be solved urgently to realize the design and preparation of high-performance carbon-based films. In order to better solve this problem, the introduction of lubricating additives may be a good choice.\[15,16\]

Graphene\[17\] has attracted much attention in macroscopic tribology applications due to its unique advantages of high chemical inertness, ultra-high strength, low shear strength, and atomically smooth surface. Currently, the tribological research of graphene major concentrates on two aspects of lubricant additives and coatings. As an additive in fluid lubrication, graphene can be added to different types of fluid lubrication systems to conspicuously improve the lubrication performance and is an ideal lubricant material for rotating/sliding contact.\[18,19\] The 3D graphene\[20\] we used is a new method to prepared 3D framework carbon (3DFC). Its comprehensive advantage is directly calcined sodium citrate without adding any additional carbon source, template and catalyst. The distance between the prepared 3DFC layers is different (0.34–0.51 nm). Because of its unique structure and more reasonable price, it promises to be an alternative to two-dimensional graphene. Moreover, studies have proved that it plays a certain positive role in tribology.\[21,22\] Hexagonal boron nitride (h-BN) called “white graphene” is a nontoxic additive that promotes the shearing of its layered structure, which consists of strong intermolecular cova-
FIGURE 1 3D gr and h-BN ethanol are used as lubricant additive, using lubricant drop casting method. A, is 3D gr ethanol solution; B, is h-BN ethanol solution (the inset is the microscopic of several ethanol solutions topography); C, is a schematic diagram of the reciprocating friction test; D, is the dispersion and stability of different additives

2 | EXPERIMENTAL SECTION

2.1 | Materials preparation

In the preparation of nanometer composite materials, 0.01 wt%, 0.1 wt% of the 3D gr and h-BN ethanol solution were prepared using anhydrous ethanol as solvent. Lanzhou Institute of Chemical Physics provided the 3D gr used in the experiment, and purchased h-BN (99.9% metals basis, 1–2 µm) from Shanghai Maclean Biochemical Technology Co., Ltd., followed by ultrasonic vibration for 60 minutes. Then the evenly mixed solution was added to the surface of DLC films, and the nanomaterial was adsorbed on the surface of the DLC film after standing at room temperature until the absolute ethanol was completely volatilized.

2.2 | Tribological test

During the whole experiment, tribological tests were performed on a ball-disc tribological wear instrument Figure 1(C) (CSM, TRN-0204015). The friction pair adopts the 6 mm GCr15 bearing steel and DLC films (316 stainless steel as the substrate). Drop casting uniform dispersing solution 0.4–0.8 mL with straw for each experiment. The test mode is reciprocating motion, the sliding frequency is 5 Hz, the stroke is 5 mm, and the time period is 20,000 Laps, the temperature and relative humidity are 30 ± 5°C and 33 ± 5% RH respectively. The normal load applied is 5 N (Hertz contact pressure is 1.07 GPa). The friction tests were carried out at room temperature, and each set of experiments was repeated three times. Then, under the condition of other parameters unchanged, the changes of friction of coefficient and wear rate of 2, 5, and 10 N under different loads are studied. For the purpose of appraise the wear rate after the test, the calculation formula is as follows:

\[ K = \frac{V}{LF} \]

where L is the total sliding distance (m) and F is the false normal load (N). For wear tracks, V wear volume (mm\(^3\)) is the product of the amplitude and area of the wear scar profile. The area of the wear track profile is measured by a 2D profiler (D-100, KLA, Tencor).

For the wear amount of ball wear scars, the formula is shown below:

\[ V = \left( \frac{\pi h}{6} \right) \left( \frac{3d^2}{4} + h^2 \right) \]
Where \( d \) is the diameter of the wear scar, \( r \) is the radius of the ball.\textsuperscript{[32]}

\[
h = r - \sqrt{r^2 - \frac{d^2}{4}}
\]

### 2.3 Characterizations

X-ray diffraction (XRD) is recorded on the copper k\( \alpha \)-ray Panalytical X’Pert PRO. Fourier infrared spectrum (FT-IR, Nicolet IS 10) of 500–4000 cm\(^{-1}\) was recorded by KBr tablet on FT-IR spectrometer. After the friction test, the optical microscope, scanning electron microscope (SEM, TESCAN MIRA3) and transmission electron microscope (TEM, Tecnai G2 TF20) were used to observed surface morphology and micro-morphology of the contact area. Raman spectrometer (Renishaw, in Via Raman Microscope) is used to characterize the bonding structure of the worn area. The content of elements in the wear scar zone was determined by X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi).

### 3 RESULTS AND DISCUSSION

3D gr and h-BN ethanol are used as lubricant additive, using ethanol solution drop casting method (Figure 1A and B). The morphology of graphene, h-BN and 3D gr/h-BN composite nanomaterials (3D gr and h-BN were mixed at a 1:1 volume) were characterized by TEM, as shown in Figure 1. We can clearly see from Figure 1 that the 3D gr and h-BN have a layered structure, and the thickness of the h-BN nanosheets is slightly thicker than that of the 3D gr. Through the 3D gr/h-BN composite nanomaterials, it can be seen that the two of them form a stacked layered structure, which indicates that the stack of h-BN and graphene nanosheets form a 3D gr/h-BN nanocomposite material. The graphene and boron nitride nanocrystals on the 3D gr/h-BN composite nanomaterials can be clearly identified. Figure 1D bottom line displays the photos of different dispersion solutions after standing 2 hours, and we can see partial precipitation of the mixed solution. A picture of the top line after 1 hour ultrasound shows that they all have good dispersion, which is a necessary condition for the 3D gr/h-BN composite nanomaterial to be used as an additive.

We first disperse 3D gr, h-BN, and 3D gr/h-BN powder uniformly in ethanol solution respectively, sonicate it for 1 hour, dry it in a vacuum drying oven, and take it out for use. The typical (002) and (100) peaks (Figure 2A) of graphene occur at 25°±2° and 43°±2° respectively, corresponding to the characteristic peaks of graphene (002) and (100). As shown in Figure 2B, the diffraction peaks corresponding to the planes (002), (100), (101), (102), (004), (110), and (112) are characteristic of h-BN powder. X-ray diffraction evidently illustrates that most of the diffraction peaks of the 3D gr/h-BN composite material can be easily marked as the phase of h-BN. Due to the relatively low diffraction intensity of graphene, the characteristic diffraction peaks
of graphene are not observed in the pattern, indicating that the h-BN and 3D gr are uniformly mixed, covering the XRD signal of 3D gr. Figure 2C shows the vibration spectrum and characteristic vibration distribution of 3D gr, h-BN and 3D gr/h-BN composite nanomaterials. H-BN has a strong and sharp absorption peak at 810 cm$^{-1}$, which is caused by the out-of-plane B-N-B bending mode; there is a very wide and strong vibration peak at 1383 cm$^{-1}$. This is caused by the B-N stretching mode in the plane, and a broad peak of about 3400 cm$^{-1}$ appears in the three curves, which is due to surface moisture and a small amount of hydroxyl groups on the edge of ethanol. The FTIR spectrum reflects that the obtained 3D gr has a large number of oxygen functional groups. The FTIR spectrum of the 3D gr/h-BN composite powder has both graphene peaks and h-BN peaks, but the graphene peak is weak, indicating that the 3D gr/h-BN composite powder contains 3D gr and h-BN. It implies the successful synthesis of 3D gr/h-BN composite nanomaterials.

As shown in Figure 3, we first conducted friction experiments on 3D gr and h-BN with different mass fractions (5 N, 5 Hz, 5 mm, 20000 Laps). It can be seen from Figure 3A and B that as the concentration increases, the friction of coefficient under h-BN lubrication shows a gradual downward trend, and the friction of coefficient of 0.10 wt% h-BN concentration after friction is lower than the friction of coefficient of 0.01 wt% concentration, indicating that the increase of h-BN concentration is beneficial to reduce the friction of coefficient. The friction of coefficient under 3D gr lubrication is opposite to that of h-BN. The friction of coefficient with the addition of different mass fractions h-BN and 3D gr respectively; D, E, is the wear rate of wear scar with the addition of different mass fractions h-BN and 3D gr respectively; C, F, is the friction of coefficient and wear rate of wear scar with the addition of 3D gr/h-BN composite nanomaterials under different loads, respectively.
coefficient of 0.01 wt% concentration is lower than the friction of coefficient of 0.1 wt% concentration, demonstrating that the increase of graphene concentration is harmful to reduce the friction of coefficient. Compared with the friction of coefficient of DLC films and single additive, the friction of coefficient of 0.1 wt% concentration of h-BN and 0.01 wt% concentration of 3D gr is the lowest. Under normal circumstances, adding lubricant can reduce the coefficient of friction. However, at low concentrations, due to insufficient supply of additives, they cannot provide limited contributions in the later stages of long-term testing. When the concentration is too high, agglomeration and accumulation may cause abrasive effects and block sliding. Therefore, the appropriate concentration selection is particularly eventful. Consequently, mixing 3D gr with a mass fraction of 0.01 and 0.1 wt% of h-BN in equal volumes, it can be noticed from the Figure 3 that the friction of coefficient of 3D gr/h-BN composite nanomaterials is the lowest, proclaiming that 3D gr/h-BN composite nanomaterials have a significant effect on reducing friction. Figure 3C tested the friction of coefficient of 3D gr/h-BN composite nanomaterials under different loads under the same lubrication conditions. The load settings were 2, 5, and 10 N, and the Hertz contact pressures were 0.79, 1.07, and 1.35 GPa, respectively. It can be seen that the friction of coefficient is the lowest at 5 N, and the friction of coefficient increases significantly at 10 N, which demonstrates that the synergistic effect of 3D gr/h-BN nanocomposite under low load is beneficial to reduce friction.

It can be noticed from the wear rate of ball wear scar in the several lubrication cases in Figure 3 (D)(E) that under 5 N load, the mass fraction of 0.01 wt% h-BN has a more conspicuous wear reduction effect than other additives. 3D gr with a mass fraction of 0.01 wt% also has a good wear reduction effect under a load of 5 N. However, under a load of 5 N, 3D gr/h-BN composite nanomaterials have the best wear reduction effect, because their wear rate is the lowest. It can be seen from Figure 3F that under the load of 5 and 10 N, the wear resistance of 3D gr/h-BN composite nanomaterials is better than DLC films, which displays that the significance of 3D gr/h-BN composite nanomaterials is used as lubricant additives lies in the higher load. Finally, the advantages of 3D gr/h-BN composite nanomaterials can be summarized as follows. On the one hand, under the loading conditions of 2, 5, and 10 N, the friction of coefficient of 3D gr/h-BN composite nanomaterials are significantly lower than that of DLC films. On the other hand, 3D gr/h-BN composite nanomaterials have better wear reduction effect under a slightly higher load. Consequently, it can be concluded that for the different loads we set, at 5 N (Hertz contact pressure 1.07 GPa), compared with other loads, 3D gr/h-BN composite nanomaterials not only have a low friction of coefficient, but also have good wear resistance. It is verified that nanomaterials of different dimensions have better effect on reducing friction and wear.

Meanwhile, we also analyzed the wear rate of the wear track, as shown in Figure 4. Figure 4A and B show that the wear rate of different mass fractions of h-BN and 3D gr has little change, but the wear rate of 3D gr/h-BN composite nanomaterials is contrast to a single additive, the wear rate increased instead. From the friction of coefficient curve in Figure 3, it can be witnessed that the 3D gr/h-BN composite nanomaterial is the lowest, and the friction of coefficient changes significantly before 5000 Laps. Therefore, in order to further explore the reasons for the increase of wear rate, we tested the friction of coefficient of the first 5000 Laps (shown in Figure 5A), and the wear track profile of the film after friction is shown in Figure 5B. Through Figure 5B, it may be observed that the depth and width of the wear track at 5000 Laps before friction is basically similar to that after friction at 20000 Laps. Therefore, it may be further speculated that the wear rate of the 3D gr/h-BN composite nanomaterials is higher than that of single additives because the friction has caused severe wear on the film in the early stage, which will lead to the increase of wear rate. Figure 4C shows the wear rate under different loads. It may be discovered that the 3D gr/h-BN composite nanomaterial has almost no change in the wear rate under high load (10 N), so we can infer 3D gr/h-BN composite nanomaterials are more suitable for high load conditions. In general, the synergistic effect of the 3D gr and h-BN under high load conditions not only has a small effect on the wear rate, but also has a good protective effect on of the steel ball surface.

Figure 6 shows the scanning electron micrographs of ball wear scar under different loads after friction. From the figure, it can be observed that the wear area of the ball surface with DLC films against the friction is larger than that of the additive 3D gr/h-BN composite nanomaterial. A large amount of debris has accumulated around the wear scar, which is likely to increase the sliding resistance rather than lubrication. The furrows of different depths on the surface of the ball wear scar of DLC films can be observed (Figure 6(A)(B)(C)), which may be caused by mechanical friction. At the same time, it is worth noting that relatively uniform and smooth patches were obtained with the lubrication of the 3D gr/h-BN composite nanomaterials, with some very shallow furrows visible only in individual wear scar (Figure 6 (D) (E) (F)). Under this condition, the wear scars become significantly smaller, contrary to the friction of DLC films. The friction product as a lubricant has been transferred to the wear scar, and the formation of the transfer film is key to obtain a low friction of coefficient, producing a smoother surface and helping to reduce the wear rate. We can also see that the SEM image of the wear
scar on the steel ball after the friction test is consistent with the related friction and wear data (see Figure 3), and once again demonstrates that the 3D gr/h-BN composite nanomaterial as an additive has excellent resisted to friction and wear properties.

In order to analyze the changes in the friction interface structure, Optical microscopy and Raman analysis of different positions of ball wear scars and wear tracks were carried out through Figure 7, which can reveal the root cause of the low friction behavior of 3D gr/h-BN composite nanomaterials. Optical microscope (Figure 7A and B) and Raman analysis (inset) show that a large amount of wear debris accumulates around the wear scar, which is covered by some wear debris. The carbon transfer film was produced on the face of dual ball wear scar, and a certain degree of graphitization occurred.[41] Different positions of the appearance of the wear scar show almost the same Raman spectrum shape, and the Raman spectrum peak at the C position of the wear scar is relatively strong. Therefore, it highlights the formation of graphite-like carbon on the surface of the wear scar after sliding friction.[28,42] As shown in Figure 7C, the sharp and split D peaks and G peaks detected in the Raman spectra on the surface of the ball wear scar indicate that the film is rubbing after adding the 3D gr/h-BN mixed additive. Graphitization occurs on the surface to form a carbonaceous transfer film. Graphite-like carbon nanostructures exist in the friction layer formed on the steel ball surface.
At the same time, it can also be observed that curve 4 in the inset of Figure 7C has a peak of h-BN (1367 cm\(^{-1}\))\(^{43-45}\), which further illustrates that the transfer film contains h-BN. In addition, the wear track is significantly smaller than DLC films, more nano materials are accumulated on the wear scar, and the surface of the wear track is smoother. The nanomaterials located at the wear tracks A, B, and C (Figure 7D) have been transferred to the surface of the ball to promote the growth of the lubricating friction layer, as confirmed by Raman spectroscopy. There is almost no wear debris in the wear track under reciprocating friction, almost all nanomaterials are transferred to the surface of the steel ball. The typical DLC peaks discovered by the Raman spectra (spots A-C) in the inset further confirm...
FIGURE 8 High-resolution TEM micrographs and mechanism diagrams. A, B, is with addition of 3D gr/h-BN composite nanomaterials before and after friction, and wear debris on steel balls respectively; C, is 3D gr/h-BN composite nanomaterials friction and lubrication mechanism.

Therefore, under the synergistic effect of the 3D gr/h-BN composite additives, friction and wear are significantly improved. In summary, it can be concluded that during the friction process of adding 3D gr/h-BN composite additives, a denser graphitized friction layer is formed on the sliding interface, resulting in low friction and low wear.

For the sake of reveal the underlying mechanism more vividly, a high-resolution transmission electron microscope TEM was used to study the structure of the 3D gr/h-BN composite nanomaterials before rubbing and the wear debris on the wear scar after rubbing, further revealing the microstructure and friction mechanism of 3D gr/h-BN composite nanomaterials. The HRTEM image in Figure 8A distinctly explains that graphene nanosheets exhibit a better layered structure through weak van der Waals action. In the illustration, we can also see the multilayer structure of 3D gr. The increase in the number of graphene layers can significantly improve its lubricating properties, because graphene with multiple layers can maintain its chemical inertness at greater normal forces. In Figure 8B, it can be obviously seen that the 3D gr and h-BN adsorbed on the steel ball after rubbing are looser than before rubbing. The widening of the layer spacing indicates that a looser transfer layer is formed during the rubbing process. Under the function of reciprocating friction, 3D gr may be deformed and cannot fully exert its lubricity, resulting in its inferior wear reduction effect in actual experiments as h-BN, which verifies the occurrence of friction-induced graphene deformation during friction. There is a transfer film on the rubbed steel ball, which contains traces of graphene and h-BN flakes.

And XPS analysis was carried out on the content of wear scar elements. The content distribution of each element as shown in Table 1 and also confirmed the existence of the 3D gr and h-BN. This indicates that 3D gr/h-BN composite nanomaterials may accumulate on the friction surface to form a thicker adsorption layer during friction, which provides better protection to the surface and prevents direct contact. Generally speaking, the graphene friction sandwich has lower shear strength when sliding, resulting in lower friction. Therefore, the decrease in friction of coefficient and wear rate may be due to the mixture of graphitized graphene and h-BN nanosheets forming a friction interface sliding on the steel ball. The existence of the friction layer effectively prevents the steel ball from contacting the DLC films surface. Thereby improving the performance under room temperature environment steel-DLC contact conditions. Figure 8C schematically reveals the lubrication mechanism of the 3D gr and h-BN nanomaterials on the DLC film at room temperature in combination with their respective advantages. At room temperature, 3D gr and h-BN material enters the friction interface, and the 3D gr/h-BN composite nanomaterial forms a thin friction layer on the appearance of the steel ball. This can provide the advantages of reducing the contact area, avoiding direct contact, and protecting the surface of the DLC film from excessive wear. Therefore,

TABLE 1 The content distribution of each element on the ball wear scar with 3D gr/h-BN composite nanomaterial

| Elements | Carbon | Boron | Nitrogen | Oxygen |
|----------|--------|-------|----------|--------|
| Atomic%  | 48.21  | 13    | 9.28     | 29.51  |
the 3D gr/h-BN composite nanomaterial, can produce a synergistic lubrication effect with DLC films as a solid lubricant according to its own low interlayer shear, which provides an example for the wider application of solid lubrication in the future.

4 | CONCLUSIONS

3D gr and h-BN ethanol solution was mixed ultrasonically to produce 3D gr/h-BN composite nanomaterials, which were used as lubricating additives under different mass fractions and different load conditions under the steel-DLC friction pairs did an evaluation. The results show that compared with DLC films, 3D gr and h-BN has lower friction of coefficients at 0.01 and 0.1 wt%, respectively. However, it is worth noting that the 3D gr/h-BN composite nanomaterials show better lubricating properties than other single additives. This is mainly due to the formation of friction interface on the steel ball. The existence of the transfer film effectively prevents the direct contact between the steel ball and the DLC films surface, improving the performance under the steel-DLC contact conditions at room temperature. This research opens up a new way for the development of advanced lubrication strategies for a new generation on oil-free tribological systems based on 3D gr/h-BN as lubricant additives under different loads.

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CONFLICTS OF INTEREST

There are no conflicts to declare.

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