Characterization and tribology performance of polyaniline-coated nanodiamond lubricant additives

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Abstract: The polyaniline (PANI)-coated nanodiamond (ND) composites were fabricated by the in situ polymerization process and dispersed in base oil as nanolubricant additives by ultrasonic-assisted dispersion. The morphology and microstructures of the nanocomposites were characterized. The in-house developed reciprocating tribo-device was utilized to conduct the tribological tests. An actual CrN piston ring–nitriding cylinder liner friction pair used in the diesel engine was employed to evaluate the effectiveness of the developed nanolubricant additives. The wear tests were conducted under conditions that were close to the actual work condition of the selected friction pair. Furthermore, the anti-scuffing performance was also investigated and the associated mechanisms were analyzed. The results show the polymerization process inhibited the agglomeration of the NDs that were well dispersed in the PANi matrix. The stable hydrogen bonding interactions and the surface confinement effect promote the dispersion of the nanocomposites in organic base oil effectively. The developed additive can improve the friction and wear performance of the ring–cylinder liner friction pair by 12–19 and 15–24%, respectively, compared with the base oil. With the increase of temperature, the effectiveness of the nanolubricant additives is enhanced. Under the oil-starved condition, the friction pairs lubricated with PANi/ND lubricants can sustain a longer stable period with a lower friction force, and the anti-scuffing time is almost three times longer. Concerning the overall tribological performance, the optimal content of the PANi/ND additive in base oil is 2 wt%.

Keywords: polyaniline-coated nanodiamond, lubricant additive, dispersion, tribology performance

1 Introduction

The incorporation of functional additives in lubricating oils is one of the effective ways to improve tribological performance [1–3]. Traditional extreme pressure anti-wear additives contain phosphorus (P), sulfur (S), chlorine (Cl) and other active groups, which mainly rely on adsorption on the friction surface or reaction with the metal surface to form a chemical reaction film to achieve anti-wear and antifriction [4,5]. However, the presence of organic compounds such as P, S and Cl will corrode parts and cause environmental pollution [6,7]. For example, the commonly used anti-oxidation and anticorrosion additive zinc dialkyldithiophosphate contains P element, which is poisoned with the three-way catalyst in the automobile exhaust converter, thus increasing the exhaust emission [8,9]. The existence of these problems has made the research on the development of environmentally friendly lubricating oil additives widely concerned [10].

Nanodiamond (ND), which refers to diamond particles with a particle size below 100 nm, has been one of the attractive nanomaterials because of its unique mechanical, physical and chemical properties [11,12]. Excellent properties such as superhard, outstanding thermal...
conductivity, chemical stability, innocuity and environmental friendliness make ND have broad application prospects in lubricating oil additives, wear-resistant surface coatings, composite materials, biomedical images, microelectronic materials and military light and high-strength materials [13–15]. However, the surface energy of nanopowder particles is high, in a thermodynamically unstable state, and they are prone to agglomeration [16,17]. Thus, the difficulty in dispersion of ND is one of the reasons that restrict the application of NDs as a lubricant additive [16,18,19]. Improving the preparation process and surface modification are the two well-known methods of preventing the agglomeration, and the latter one is more prevalent. The surface modification methods mainly include surface chemical modification, mechanochemical modification, high-energy surface modification, etc. [16]. The agglomerated particles can be crushed under the action of mechanical energy, but after the external force is removed, the particles in the system are prone to re-agglomeration. If the nanoparticles are coated with some stable polymer before their agglomeration, the separation of the nanoparticles may be achieved.

Some new methods should be proposed. And studies have proved that adding carbon fiber, glass fiber and micron particles into the polymer matrix can significantly improve the bearing capacity of the polymer matrix and improve its wear resistance [20–22]. Therefore, in this study, the surface chemical modification method was used to coat the ND surface with lipophilic polymer in order to obtain the powder structure which can be stably and evenly dispersed in lubricating oil.

Polyaniline (PANI) is a common and easy-to-obtain macromolecule polymer, which attracts wide attention due to its easy synthesis and good high temperature resistance. Chen et al. [23] obtained polyaniline/nanodiamond (PANI/ND) composite microspheres by in situ emulsion polymerization, which proved that chemical bonding exists between ND particles and PANI molecular main chain and enhanced the thermal stability of PANI with good ND dispersion. Thus, PANI may be a suitable polymer that can be used to coat NDs. Chen et al. [24] also found that PANI/ND structures have excellent electromagnetic absorption properties. Moreover, it has been reported that the PANI/ND nanocomposite can enhance the corrosion resistance of epoxy coating [25]. The PANI/ND may also be an effective additive in lubrication oil.

This work aims to determine the effect of PANI/ND additives on the tribological performance, and validate the applicability of the nanocomposite in actual friction pair working under a severe working condition. The PANI-coated ND composites were fabricated by the in situ polymerization process and ultrasonically dispersed in base oil as the lubricant additive. The microstructure of nanocomposites was characterized by X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). The morphologies were observed by scanning electron microscope (SEM) and transmission electron microscope (TEM). The friction and wear as well as the scuffing tests were conducted to evaluate the effectiveness of the PANI/ND additives. Finally, the associated wear behaviors were discussed.

2 Experimental apparatus and procedure

2.1 Fabrication of the PANI-coated ND composites

The molecular model of ND dispersed in base oil is shown in Figure 1. First, the ND particles are well dispersed due to the interactions between the aniline cations and ND, as well as the following in situ polymerization, forming

![Figure 1: Molecular model of ND dispersed in base oil.](image-url)
stable PANi/ND composites. Then, the PANi/ND composites are dispersed with base oil as lubricant by ultrasonic-assisted dispersion. The specific steps are as follows:

First, 0.2 mL aniline monomer (Macklin, >99.5%, Shanghai) was added into 20 mL of 1 N dilute hydrochloric acid solution, mechanically stirring for 30 min at 0–5°C, 500 rpm. Next, 24 mg ND (XFNANO, >97%, Nanjing, the particle size of the NDs ranges from 5 to 10 nm which was provided by the supplier) was then added into the above aniline cationic solution under 0–5, with 1 h ultrasonication. Then, 0.456 g ammonium persulfate (China Pharmaceutical Group Chemical Reagent Co., Ltd.) dissolved in 50 mL of dilute hydrochloric acid was dripped slowly and uniformly into the above solution, and stirred under 0–5°C for 4 h. The reaction solution was then kept for 16 h to finish the polymerization. The samples were separated and purified using a centrifuge (H1750R, Hunan Xiangyi Laboratory Development Co., Ltd.). Then, the product was washed and dried to obtain the PANi/ND. The base lubricants used in this study are commercial synthetic oils (HVI-5 base oils). PANi/ND-modified lubricants were obtained by adding 1, 2 and 3 wt% PANi/ND into HVI-5 base oil, respectively.

2.2 Microstructure characterization of the PANi-coated ND composites

The SEM (Zeiss SUPRA.55.SAPPHIRE, Germany) and TEM (JEOL JEM-100CX, Japan) were employed to observe the morphology of the PANi/ND composites. XRD (Rigaku D/Max-Ultima X-ray diffractometer, Cu Ka radiation) was used to characterize the internal orientation and order degree of the PANi/ND composites in the range of 5–75° at 4°/min. FTIR (Bruker TENSOR II spectrometer, 400–4,000 cm⁻¹, with 4 cm⁻¹ resolution) was performed to analyze the functional groups, element composition and hybridization on the surface of PANi/ND composites.

2.3 Tribological tests

2.3.1 Block-on-Ring test

The friction and wear properties of PANi/ND composites with different content in lubricating oil were tested by using the block-on-ring tribology tester. Both the ring and block specimens were made of GCr15 steel. The testing configuration is shown in Figure 2, the rotation ring has a dimension of Φ50 mm × 25 mm with the surface roughness of 0.347 μm and hardness of 1,169 HV0.1. The fixed block is much smaller with the size of Φ10 mm × 10 mm, its smoother (0.112 μm) and harder (1433.7 HV0.1) than the block. The friction pairs were subjected to the loading of 50 N and the testing temperature was 70°C. The frictional force during the sliding can be recorded and then calculated into coefficient of friction accordingly. The morphology of the wear marks on the blocks was observed by an optical microscope OLYMPUS GX51 (Olympus, Japan) and the wear amount was characterized by measuring the width of the wear scars on the surface.

2.3.2 Friction and wear test under intensified loading and oil-starved conditions

In order to validate the applicability of the nanolubricant additives in actual friction pair and determine their

![Figure 2: The configuration of the block-on-ring test and the testing specimens.](image-url)
effectiveness under different temperatures, the real piston ring (CrN-coated piston ring provided by CYPR ASIMCO Shuang Huan Piston Ring Co., Ltd) and cylinder liner (nitriding cylinder liner provided by Chengdu Yinhe Power Co., Ltd) friction pair used in diesel engine was selected. The wear tests of the ring and liner pair were conducted under the condition that was close to the top dead center of the combustion chamber in diesel engine, but the loading was intensified to accelerate the wear process. To further investigate the effect of the nanolubricant additives on the wear performance under the lubricant starvation condition, the scuffing tests were performed as well. The testing parameters for the wear and scuffing tests are listed in Tables 1 and 2. The details of the tribo-tester were reported in our previous work [26]. After wear tests, the wear losses of the ring and liner specimens were measured by the LEXT OLS4000 confocal laser scanning microscopy (CLSMA, Olympus, Japan). The method of wear loss measurement was identical with the study reported previously [27]. The worn surface morphology of the liner specimen was examined by field emission SEM (Zeiss SUPRA 55 SAPPRIRE, Germany). For each nanolubricant additive concentration, both wear and scuffing tests were carried out four times.

3 Results and discussion

3.1 Morphology and microstructures of the PANi-coated ND composites

From the SEM image of Figure 3(a) and the TEM image of Figure 3(b), it can be seen that the morphology of ND is irregular spherical with severe agglomeration. From the high-resolution transmission image of Figure 3(c), the interlayer spacing is 0.210 nm, corresponding to the [111] crystal surface of ND [28]. It can be seen from the SEM image in Figure 3(d) that the prepared PANi/ND composite presents as irregular globular aggregate. The size of the small protrusions onsite the aggregate (Figure 3(e)) matches well with that of the ND, indicating that the ND was well preserved after the polymerization and well dispersed in the matrix, which can also be clearly seen from the TEM image (Figure 3(f)).

Figure 4 shows the XRD comparison of ND and PANi as well as the PANi/ND composite. As for PANi/ND and ND, the characteristic peak of ND could be found at 2θ = 43.5°, which corresponds to the (111) crystal plane of the ND face-centered cubic lattice (diamond, JCPDS 06–0675). The characteristic peak of PANi at 2θ = 8.7°, 15.5°, 20.6° and 25.5° are for (001), (011), (020) and (200) planes of PANi, respectively [28,29]. The XRD results further indicate that the ND particles are well coated and preserved by PANi in the PANi/ND composites.

It can be seen from the FTIR spectra (Figure 5) that the relative intensity of carbonyl peak at 1,635 cm⁻¹ of ND decreases obviously after the combination with PANi, indicating that there are hybridization or coating effect between ND and PANi. The characteristic peaks of PANi could be found in the FTIR spectrum of PANi/ND with some certain red or blue shifts compared with those of PANi. The 1,492 cm⁻¹ band in PANi (C–C) stretching vibration in benzenoid) red shifted to 1,486 cm⁻¹ for PANi/ND, which indicates the formation of hydrogen bond between PANi and ND [30,31]. On the other hand, the 1,296 cm⁻¹ band (C–N–C stretching vibration) blue shifts to 1,306 cm⁻¹, which should mainly due to the surface confinement effect of ND; that is to say, the PANi

| Table 1: The parameters of the wear test |
|----------------------------------------|
| Period | Parameters | Lubricant amount (mL/min) |
|--------|------------|--------------------------|
| Running-in | 100, 150, 200°C; 10 MPa; 1 h | 0.1 |
| Experiment | 100, 150, 200°C; 40 MPa; 6 h | 0.1 |

| Table 2: The parameters of scuffing test |
|-----------------------------------------|
| Period | Parameters | Lubricant amount (mL/min) |
|--------|------------|--------------------------|
| Low load running-in | 120°C, 10 MPa, 10 min | 0.1 |
| High load running-in | 190°C, 20 MPa, 150 min | 0.1 |
| Fuel cut | 190°C, 20 MPa, to scuffing | 0 |
chains will be limited and oriented along the ND surface [24,32], increasing the vibrational steric hindrance of the corresponding group.

Based on the structural analysis, it could be proved that ND could be well dispersed in PANi, forming stable hydrogen bonding interactions. Meanwhile, given to the surface confinement effect of ND, PANi is effectively tied and coated on the surface of ND, which could well prevent the re-agglomeration of ND and promote its dispersion in organic base oil. Thus, as an additive to lubricating oil, PANi/ND may have some influence on antifriction and wear properties.

3.2 Effect of the nanolubricant additive content on the tribological performance

Figure 6 shows the results of the block-on-ring tests lubricated with different nanolubricant additive content. It can be seen from Figure 6(a) that the addition of 2 wt% PANi/ND nanocomposite results in a relatively lower friction force and shorter running-in period. The calculated friction coefficients first decrease and then increase with the nanocomposite content increase, as shown in Figure 6(b). The wear loss of the block specimen shows the similarity in trend with friction coefficient variation (Figure 6(c)), implying that the PANi/ND composite is beneficial to improve the friction and wear properties. It is interesting that the antifriction effect of the nanocomposite additives becomes worse when the concentration is 3 wt%.
It may be attributed to the agglomeration of the ND with a higher concentration. The agglomerated nanoparticles with larger particle size destroy the integrity and continuity of the oil film, and bring in the scratching and abrasive wear [33]. Moreover, the agglomerated NDs may be too big as compared to the gap between asperities; they will not be able to deposit on the contact zone which will lead to poor lubrication. Thus, the antifriction effect is suppressed. Therefore, the optimal PANi/ND additive content in base oil can be determined at 2 wt% in this study.

### 3.3 Effect of the temperature on the tribological performance of nanolubricant additives

Figure 7 shows the typical friction force curves of the nitriding liner and the PVD-CrN piston ring in wear tests lubricated with different nanolubricant additives content. At 100°C (Figure 7(a)), after the running-in period, the friction pair enters the high-load wear stage, in which the friction force fluctuation gradually decreases, implying that stable wear is reached. It can be found that the friction force is slightly lower when the PANi/ND additive concentration is 2 wt%; the other three friction pairs show no significant difference in friction force in the late stage of stable wear. Figure 7(b) illustrates the change of friction force with time at 150°C for each friction pair; the overall variation trend is similar to that of 100°C, but the level of friction force for each pair has a certain increase. The friction force corresponding to the base oil fluctuates continuously and presents an upward trend with time, even around the end of the test. In contrast, the other three pairs lubricated with the ND composite additives still show the stable wear and lower friction force compared with the base oil, and the lowest friction force is observed when the nanocomposite content is 2 wt%. The phenomena are consistent with the results obtained by the ring block test. When the temperature increases to 200°C, the friction reduction effect of the PANi/ND additive is more noticeable (Figure 7(c)). At this temperature, the friction force of the base oil is considerably larger than the nanocomposite lubricated pairs. The nanocomposite content of 2 wt% results in the
lowest friction force as well, and about 15% reduction of friction compared with the base oil lubricating can be obtained.

Figure 7(d) shows the summarized friction coefficients of the friction pairs lubricated under different PANi/ND addition. It can be seen that the overall friction force increases with the testing temperature and the PANi/ND additive has a friction-reducing effect for the base oil. The reason is that the evaporation rate of lubricating base oil becomes faster when heated, and its viscosity decreases with the increase of the temperature. At this time, the lubricating oil film becomes thinner, the contact probability of asperities becomes greater. As a result, the proportion of boundary lubrication dominates the friction interface, causing a larger direct contact of the friction pair, and the friction coefficient increases accordingly. When the PANi/ND additives were added in the base oil, it will play a role under the boundary lubrication state. The micro-polishing effect of NDs may result in a smoother interface, because the roughness of the lubricating surface can be reduced by ND-assisted abrasion. On the other hand, the hard spherical ND particles between the friction pairs can separate the contact of asperities effectively when the oil film is thin, so that the contact state between the friction pair is most likely changed from “sliding friction” to “rolling friction.”

Third, the nanoparticles in the lubricating oil have the ability to fill scars and grooves of the friction surface, bringing the surface mending effect [34–36]. Therefore, the friction force diminution can be obtained. Comparing the variation of friction coefficients at different temperatures, it is found that the nanocomposite lubricant has little effect, but when the temperature rises to 200°C, the friction coefficient between the friction pairs is obviously distinguished, indicating the friction reduction effect of the nanocomposite is more apparent as the temperature is higher.

Figure 8 shows the wear losses of the nitriding liner and the PVD-CrN piston ring in wear tests lubricated with different PANi/ND additives. It can be seen that the wear loss of the cylinder liner is lower than that of the piston ring, which is caused by the relatively low hardness of the piston ring (cylinder liner 1,109 HV0.1, piston ring 705 HV0.1). As the piston ring can be replaced during maintenance, the friction pair of the cylinder liner and piston ring is usually designed with better wear resistance of the cylinder liner than that of the piston ring. As the temperature increases, the wear losses of both liner and ring specimens keep increasing. This is also because the high temperature deteriorates the lubrication state. For the nitriding cylinder liner (Figure 8(a)), the addition of nanocomposite lubricant at 100°C has almost no effect on the wear loss of the cylinder liner specimen. However, when the operating temperature rises to 150 or 200°C, the nanocomposite lubricant oil corresponding to the wear loss of the liner specimen lubricated with
PANI/ND additives is lower than that of the base oil, and the wear reduction effect of the nanocomposite lubricant is more apparent when the concentration of the nanocomposite lubricant is 2 wt%, especially for the higher temperature. Figure 8(b) shows the wear loss of PVD-CrN piston rings tested under different temperatures and lubricants, presenting the similar trend to the wear loss of the nitriding cylinder liner, but the wear depth of the piston ring under nanocomposite lubricant is constantly lower than that of the basic regardless of the temperature. At each temperature, the minimum wear loss is always found for 2 wt%. It can be concluded that the PANI/ND additive can significantly improve the anti-wear performance of the selected cylinder liner and piston ring friction pair compared with the base oil, especially at a relatively higher temperature.

Figure 9 shows the wear surface morphology of the liner specimens under lubricants at 200°C. It is found that the base oil lubrication causes the relatively severe wear, and some of the honing pattern has disappeared, but...
there is no typical adhesive or abrasive wear characteristic, such as plastic deformation, peeling off or furrow. The wear of the liner specimen lubricated with 1 or 3 wt% nanocomposites is more gentle compared with the base oil condition, but the wear surface under the 2 wt% PANi/ND additive lubrication basically keeps the initial as-machined appearance of the cylinder liner, and the honing pattern is relatively clear, indicating that the slight wear and least wear loss in all the tests, indicating the antifriction and anti-wear effect of the nanocomposite lubricant.

3.4 Effect of the nanolubricant additives on the wear performance under the lubricant starvation condition

Figure 10 shows the typical friction force curves of the nitriding liner and the PVD-CrN piston ring in oil-starvation tests lubricated with different PANi/ND content. After running-in stage, the lubricating oil can be evenly dispersed on the contact surface. Then, the supply of lubricating oil is cutoff (about 9,600 s), and the friction pairs fall in the oil-starved lubrication stage. It can be seen that the friction force corresponding to base oil lubrication increases sharply 40 min after the oil cut, implying that scuffing occurs. In contrast, the other three groups of friction pairs lubricated with PANi/ND lubricants sustain a long stable period with a relatively low friction force, resulting in a longer anti-scuffing time which is almost three times than the pair tested in the base oil. Similarly, the best anti-scuffing performance is observed when the nanocomposite lubricant concentration is 2 wt%. Under the oil-starved condition, the lubricant additives and the oil retention capacity of the friction pair determine the anti-scuffing performance. Compared with the base oil condition, the NDs in the nanocomposite can reduce the interfacial friction force and the generation of frictional heat, as a result, the consumption of the stored lubricating oil can be inhibited. On the other hand, the PANi has a relatively good heat resistance [37,38] and will play a role of solid lubricant when heated. The softened PANi is a kind of viscous polymer liquid, which shows the low shear stress and can further reduce the friction force and prevent the direct contact of the asperities on the friction pair surface.

The SEM images of the nitriding cylinder liner surface after scuffing tests are illustrated in Figure 11. The morphology of the scuffed liner surface can be identified as the scuffing zone and the normal wear zone. For the morphology obtained under the condition of base oil (Figure 11(a)), the scuffed area is severely damaged, and the honing pattern is almost worn off, while the honing pattern in the normal wear area is still visible. As the white bright layer on the surface of the nitrided cylinder liner is an intermetallic compound and shows the high strength and poor affinity with the CrN piston ring, no peeling-off, the typical scuffing feature, is found. Regarding the scuffed morphology under the

![Figure 10: The typical friction force variation of the nitriding liner and the PVD-CrN piston ring in scuffing tests lubricated with different PANi/ND additives.](image-url)
2 wt% nanocomposite lubrication, there are only a few scratching bands in the scuffing zone, and the corresponding area is also much smaller than the base oil condition (Figure 11(b)), but the scratching area still shows severe wear. The edges of the bearing platforms between the honing patterns deform plastically, and the honing pattern almost disappears due to the filling of plastic deformation and wear (Figure 11(c)). However, the normal wear area is worn very lightly and almost maintains the original morphology of the cylinder liner surface. In conclusion, the PANi/ND additive can improve the resistance to adhesive wear and scuffing under oil-starved conditions.

4 Conclusion

The PANi-coated ND composites were fabricated by the in situ polymerization process and ultrasonically dispersed in base oil as the lubricant additive. The developed additive is basically the nanocomposite material. This work proposed a novel nanocomposite additive, and its tribological performance was evaluated by the real piston ring–cylinder liner pair under the conditions that were close to the actual working conditions of the selected friction pair. Thus, the applicability of PANi/ND nanolubricant additive in diesel engine was validated to a certain extent. The conclusions were drawn as follows:

- The NDs were well preserved after the polymerization and uniformly dispersed in the base oil, resulting in the nanolubricant additives. The stable hydrogen bonding interactions provide the surface confinement effect of ND; thus, the PANi can be effectively tied and coated on the surface of ND, which could well prevent the re-agglomeration of ND and promote its dispersion in organic base oil.
- The developed additive can improve the friction and wear performance of the ring–cylinder liner friction pair by 12–19 and 15–24%, respectively, compared with the base oil. With the increase of temperature, the effectiveness of the nanolubricant additives is enhanced. There is no typical adhesive or abrasive wear characteristic when the nanocomposites were added in the lubricant. However, the beneficial effect of tribological performance disappears when the additive concentration is 3 wt%.
- Under the oil-starved condition, the friction pairs lubricated with PANi/ND nanolubricant additive can sustain a longer stable period with a lower friction force than the pair tested in the base oil. The anti-scuffing time is almost three times longer. With respect to overall tribological performance, the optimal content is 2 wt%.
In the future, the fabrication of the PANi/ND nanolubricant additive will be optimized by modifying the polymerization process, changing the particles size of the NDs and adjusting the molecular weight of the PANi, and the expected reduction percentages of friction coefficient and wear loss reduction compared with base oil can probably reach 25 and 30%, respectively.

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