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Anti-wear properties evaluation of frictional sliding interfaces in automobile engines lubricated by copper/graphene nanolubricants

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Abstract: Owing to the significance of improving fuel economy, reducing emissions, and extending the durability of engine components, this study focused on the tribological performance of nano-additives. In this study, copper (Cu) and graphene (Gr) nanomaterials were dispersed in a fully formulated engine oil (5W-30) with different concentrations. The tribological trials were investigated under various speeds and loads, utilizing a reciprocating tribometer to mimic the ring/liner interfaces in the engine. The frictional surface morphologies were comprehensively analyzed using electron probe X-ray microanalysis (EPMA), field emission scanning electron microscopy (FESEM), energy dispersive spectrometer (EDS), and three dimensional (3D) surface profilometry to explore the mechanisms responsible for improving the tribological performance of the frictional sliding parts in the engine. The tribological test results illustrated that lubrication by nano-additives reduced the wear rate (WR) and friction coefficient (COF) by 25%–30% and 26.5%–32.6%, respectively, as compared with 5W-30. The results showed that this is a promising approach for increasing the durability and lifespan of frictional sliding components and fuel economy in automobile engines.

Keywords: engine tribology; nanomaterial; nanolubricant; friction; wear; tribofilm

1 Introduction

The current challenges in automobiles engines for improving the tribological performance and extending the durability of frictional sliding components require novel lube oils that readjust to various operating circumstances [1–3]. 90% of the lube oils sold commercially compose of hydrocarbon molecules, and the rest are additives that govern performance [4]. Therefore, many researchers have studied different technologies for exploring novel methods to replace environmental harmful additives that cause adverse emissions (zinc dialkyldithiophosphate) and other additives that include sulfated ash, sulfur, and phosphorous without compromising on tribological engine behavior with eco-friendly additives, such as nanomaterials and ionic liquids [5, 6]. The total frictional power losses within different sliding contact interfaces contributed 20% of the overall losses within automobile engines [7, 8]. Consequently, an improvement in the engine tribological performance serves to improve efficiency and fuel economy, especially the tribological performance of the ring/liner interfaces [9].

Over the past few years, rapid progress in the development of nanolubricant additives that rely on nanoparticle mechanisms has been made, such as the formation of a protective layer on surfaces and the creation of a rolling influence between sliding surfaces
for energy savings and emission reduction [1, 10–14]. Ali et al. [15] studied the change in the friction characteristics for the ring-liner contact with the crankshaft angle using TiO$_2$ and Al$_2$O$_3$ nanomaterials in 5W-30. The tribological test results showed that nanolubricants are more efficient at the top and bottom dead center of the stroke (the top dead center (TDC) and the bottom dead center (BDC) locations) during boundary/mixed lubrication. The friction coefficient (COF) was reduced by 50% and 45% for both the TiO$_2$ and Al$_2$O$_3$ nano-additives, respectively. Li et al. [16] investigated the influence of ZrO$_2$/SiO$_2$ nanoparticles with base oil on the tribological properties. The results illustrated that the COF decreased 16.24%, utilizing 0.1 wt% concentration. Another investigation by Ali et al. [17] investigated the effects of TiO$_2$, Al$_2$O$_3$, and TiO$_2$/Al$_2$O$_3$ nanoparticle (8–12 nm) additive into 5W-30 on the thermophysical parameters. The results showed that the viscosity index and thermal conductivity of the TiO$_2$/Al$_2$O$_3$ nanolubricants improved by 2% and by 12%–16%, respectively, compared to that of the lubricant without nanomaterials. Furthermore, Li et al. [18] reported a 14% improvement in the thermal conductivity of ethylene glycol (EG) when the ZnO nanoparticles was added with 30-nm diameter and 10.5 wt% concentration.

The effectiveness of the nanolubricants not only depends on the type of nanoparticles but also on their morphology. The results by Dai et al. [6] confirmed that the majority of the nanolubricants consist of metals, metal oxides, and sulfides. Furthermore, the nanomaterial morphologies can be spherical, sheets, or nanotubes. The spherical shape of the nanomaterials offered superior anti-friction properties. The reason is strongly related to the rolling mechanisms between the rubbing surfaces during the sliding process [19, 20]. The nanolubricants themselves are excellent as a self-repairing function due to the formation of a protective layer that is deposited at the contact area between the surfaces of the asperities [21]. The self-lubricating replenishment is described as a self-coating film resulting from the friction process (chemical reactions), which is deposited on the worn surfaces but has a different construction and chemical composition [15, 22–24]. Based on the experimental tests by Padgurskas et al. [25], the Cu nano-additives are more effective in mixed and boundary lubrication than in full film lubrication. This indicates that the potential interaction of the rubbing surfaces is necessary for the formation of Cu tribofilm and its tribological performance.

In summary, as can be observed from these prior studies, the nanoparticles are eco-friendly and economical when used as nano-additives inside the base lube oils. However, few studies have focused on the major mechanisms serving to improve the tribological behavior of the piston ring-cylinder liner contact in the engines. In this research, the aim is to provide the main reasons and explanations how nanolubricants can assist in extending the durability of the frictional interfaces in automobile engines under different circumstances.

## 2 Experimental

### 2.1 Materials

Cu and Gr nanomaterials were employed as nano-lubricant additives inside a fully formulated commercial engine oil (5W-30) to illustrate the influence of the nano-additives on the wear and friction behavior under various sliding speeds and contact loads. The Cu and Gr nanomaterials were purchased from XFNANO Company, China.

### 2.2 Tribometer and experimental procedures

The tribological properties of the Cu and Cu/Gr nanolubricants were evaluated using a reciprocating tribometer to mimic the motion of the ring/liner interfaces in accordance with the ASTM G181-11 [26]. Tribometer and rubbing specimens used in the tribological tests were displayed in Fig. 1. In this set-up, the liner is sliding against a stationary piston ring. The details of the tribometer and their properties were previously described in our earlier works [15].
The COF was calculated \textit{in situ} by measuring the frictional force (using a piezoelectric sensor) that was then divided by the applied load while the wear rate (WR) of the rubbing samples in mm$^3$·N$^{-1}$·m$^{-1}$ was quantified by measuring the wear volume (using a 3D profilometer, Nanovea ST400). Then, it was normalized by the normal load and sliding distance. The details of the measurement technique were explained by Truhan et al. [27]. The WR of the ring and liner was determined using Eq. (1).

$$\text{WR} = \frac{\text{Worn volume}}{\text{Applied load} \times \text{Sliding distance}}$$

The tribological tests were carried out at different contact loads between 90–368 N (corresponding to a contact pressure of 1.95–7.9 MPa) and sliding speeds from 0.154 to 0.6 m/s under 100 ± 3 °C temperature to mimic TDC location near the surface of the liner temperature [27]. Furthermore, the applied loads were chosen to simulate the nominal contact pressure between the ring and liner during combustion at 50% of maximum engine load during actual engine operation [28]. Based on the Hamrock and Dowson equation [29], which depended on the material parameters and surface roughness measurements, the estimated lambda ratio did not exceed 0.87, confirming that the contact locations were inside the boundary lubrication system. Each friction test was carried for the duration of 25 min. The same amount of lubricant was utilized during all experiments for the estimation of friction and wear (6 ml). At least three trials were performed for each lube oil type. Furthermore, the rubbing specimens were allocated for each point in the experiment for both the reference oil and nanolubricants to obtain reliable data from the friction tests. Before the tribological tests, the rubbing samples were ultrasonically cleaned for 15 min in acetone and completely dried.

### 2.3 Worn surfaces examination

The crystalline structures and phases of the nano-additives were determined by X-ray diffraction (XRD, D/MAX-RB, RIGAKU Corporation, Japan) using Cu Kα radiation at 30 kV and 40 mA at a scanning speed of 0.01 (°)/s. The Vickers hardness of the frictional samples (ring and liner) was measured using the HVS-1000 Vickers hardness instrument (Beijing Times Peak Technology Co., Ltd., Beijing, China). Following the sliding tribological tests, the morphologies of the rubbing surfaces of the ring and the liner samples were analyzed by various techniques, such as electron probe microanalysis (EPMA, JXA-8230, JEOL Corporation, Japan), field emission scanning electron microscopy (FESEM, ULTRA-PLUS-43-13, Zeiss Corporation, Germany), energy dispersive spectroscopy (EDS, Inca X-Act, Oxford Instruments, Britain), and 3D optical profilometry (ST400, Nanovea, America), in accordance with ISO 25178.

### 3 Results and discussion

#### 3.1 Nanolubricant characterization

Figures 2(a) and 2(b) illustrate the morphology of the Cu and Gr nanomaterials. The Cu nanomaterials have sizes ranging from 10 to 20 nm, while the Gr nano-plates have a diameter and thickness of 5–10 μm and 3–10 nm, respectively. The crystal nanostructure of the nanomaterials was examined through XRD analysis. Figure 2(c) presents the XRD pattern of the Cu and Gr nanomaterials. The diffraction of the Cu peaks is located at $2\theta = 43.39^\circ$ and $50.49^\circ$ which can be indexed to the (111) and (200) planes of metallic Cu. These peaks were very compatible with the standard JCPDS Card No. 04-0836, which confirmed the pure...
metallic Cu and cubic characterizations. Moreover, there are other diffraction peaks that appeared at $2\theta = 29.65^\circ$, $36.53^\circ$, $42.43^\circ$, $61.56^\circ$, $73.57^\circ$, and $77.39^\circ$, corresponding to the (110), (111), (200), (220), (311), and (222) planes, respectively, confirming the formation of Cu$_2$O nanocrystals. The observed Cu$_2$O peaks are listed according to JCPDS No. 05-667 [30]. In summary, the XRD pattern of the Cu nanomaterials showed two crystalline phases, which are metallic Cu and Cu$_2$O, and there is no other phase of copper oxide (CuO). Meanwhile, from the Gr XRD pattern, it can be noted that strong diffraction peaks appeared at $2\theta = 26.381^\circ$ and $54.542^\circ$ in correspondence with the crystal planes of (002) and (004) of hexagonal graphite, respectively [10]. Consequently, these peaks clearly verified that the crystalline structure of Gr was intact.

The nanolubricant samples are stirred using a magnetic stirrer for four hours to mono-disperse the nanomaterials into the reference oil. The dispersed nanolubricant samples are monitored for 11 d using UV–vis analysis at different times. Figures 3(a) and 3(b) display the proposed samples of the Cu and Cu/Gr nanolubricants in which the preparation of the samples included four concentrations (0.03, 0.2, 0.4, and 0.6 wt%) and 2 wt% of oleic acid (OA) as a solvent to help in the nanomaterial dispersibility into the baseline oil. Fully formulated engine oil (5W-30) was used as a baseline lubricant to exhibit the effects of Cu and Gr nano-additives as friction modifiers. The kinematic viscosity for the reference oil and Cu/Gr hybrid nanolubricants for various concentrations under temperatures of 40 and 100 °C is presented in Table 1.

The kinematic viscosity of the oils was estimated according to the GB/T 265-1988 standard at Wuhan
Table 1  Kinematic viscosity of the Cu/Gr hybrid nanolubricants.

| Lubricant type | Concentration (wt%) | Kinematic viscosity (mm²·s⁻¹) |
|---------------|---------------------|-------------------------------|
|               |                     | 40 °C                         | 100 °C                        |
| Base oil      | 0                   | 54.06                         | 9.42                          |
| Cu/Gr         | 0.03                | 54.30                         | 9.40                          |
| nanolubricant | 0.2                 | 54.80                         | 9.70                          |
|               | 0.4                 | 55.00                         | 9.90                          |
|               | 0.6                 | 55.20                         | 10.00                         |

Runjia Lubrication Products Testing Consulting Co., Ltd. Compared with the baseline lubricant, the kinematic viscosity of the Cu/Gr nanolubricants containing various concentrations at 40 and 100 °C produced a slight increase in the viscosity values. Consequently, the small variation in the viscosity could assist in confirming the effective role of the Cu/Gr nano-additives in enhancing anti-wear properties during the friction process. These results are in agreement with those obtained with lube oil (5W-30) containing Gr nano-additives [10].

To check the stability of the Cu and Cu/Gr nano-additives, UV−vis spectroscopy was used to elucidate the stability of the nanolubricants. The stability of the formulated nanolubricants was monitored at a wavelength of 482 nm (λmax) for 11 d, as shown in Fig. 3(c). The higher peak of absorbance implies a better dispersion of nano-additives within the base oil. The UV results show that the dispersibility of the nanolubricants showed satisfying stability for 11 d after the mixing. Notably, the nanolubricant stability decreased with increasing storage time because of the sedimentation of the nano-additives into the base oil. Aggregation of the nano-additives occurs whenever the Brownian motion and van der Waals attractive forces of the nanomaterials are greater than the repulsive forces, based on the theory developed by Derjaguin, Landau, Verwey, and Overbeek (DLVO theory) [15]. Further investigation is needed to study the factors affecting the dispersion stability.

3.2 Tribological performance of nanolubricants

To determine the optimum concentrations of nano-additives, the COF was measured for various concentrations of Cu and Cu/Gr nanoparticles (0.03, 0.2, 0.4, and 0.6 wt%). Figure 4 shows the average COF for these concentrations under a 216 N load and 0.25 m/s sliding speed. The error bars indicate the standard deviations, which are calculated using the OriginPro program. Based on the friction results, it is demonstrated that the COF for all the Cu and Cu/Gr nanolubricant samples was less than that of the reference oil (5W-30). Moreover, it is evident that the Cu and Cu/Gr nano-additives with the concentrations of 0.4 wt% were the best samples of nanolubricants. This might be caused by the saturation of the contact area between the liner and ring surfaces with nano-additives during the dominance of the boundary lubrication regime. In this case, the nanoscale dimension and 0.4 wt% concentration can help the nano-additives fill the valleys within asperities, leading to a reduced COF. In 0.6 wt% concentration, the agglomeration of the nanomaterials is likely to occur in the reference oil, which can be higher than the oil film thickness in the contact region. In this case, the nano-additives play a role as a spacer in reducing the metal contact between the asperities and lead also to the decline in the COF as compared with the reference oil [31, 32]. Accordingly, the optimum concentration of the nano-additives was 0.4 wt%, which is then used in the next tribological tests and compared with the reference oil. Furthermore, the addition of OA only without nano-additives decreased the COF by 9% at a concentration of 2 wt% in the reference oil. The decrease in COF was not due to physical adsorption of OA on rubbing surfaces but rather to its chemical reaction, as presented by another study [33].

![Fig. 4](http://friction.tsinghuajournals.com)  Effect of the Cu and Cu/Gr nano-additives concentration on the COF. The error bars indicate the standard deviations.
One of the distinguishing features of the piston ring-cylinder liner contact is that the lubrication mechanism occurs with different types of lubrication (boundary, mixed, and hydrodynamic), which occur simultaneously in the reciprocating single stroke (one wear track). Figure 5 shows the real-time COF of the reference oil (5W-30), Cu, and Cu/Gr nanolubricant samples using 0.4 wt% nano-additive concentration. The test contact load of the contact load was 368 N, and the average reciprocating sliding speed was 0.25 m/s. The variation in the COF with operating time, described by the negative part of the trend is due to the change in speed direction through the reciprocating motion. The friction mechanism is different during one stroke because of the various lubrication systems that can affect the one stroke [5]. The friction behavior revealed that the highest COF was detected at the TDC and BDC of the stroke, due to the low sliding speed, which becomes instantaneously zero at TDC and BDC and limits adequate passage of the lubricant to these positions, resulting in greater metal contact between the frictional interfaces (boundary lubrication). The lowest COF was at the middle of the stroke because of sufficient lubricant entry (hydrodynamic lubrication) as a result of the highest sliding speed [17]. The results explained that the boundary friction coefficient at TDC and BDC declined by 33% with the use of Cu/Gr nanolubricant, as compared with the reference oil. Nano-additives are the most efficient in the boundary lubrication system.

The average COF results for both the Cu and Cu/Gr nanolubricants and reference oil versus the applied loading and average sliding speed are shown in Fig. 6. The results explained that the average COF is decreased slightly for both the nano-additives and base oil, following the increase in the speed and load. The principal reason for this reduction may be the significant contact pressure over the asperities under elevated loads, causing a reduction in the boundary COF. In addition, the decrease in the COF with the increase in sliding speed may be due to the increased momentum transfer in the normal direction with increasing sliding speeds, generating an upward force on the top rubbing surface [5]. These results enhanced the separation between the rubbing surfaces, which will reduce the real contact area. Consequently, the metal contact for asperity deformation was reduced, resulting in a decline in the COF. Furthermore, the frictional heating of the asperities can also accelerate the oxidized layers formed on the worn surfaces with the increase in sliding speed. The results also indicated that the the average COF of the nanolubricants containing Cu and Cu/Gr nanomaterials decreased versus contact loads and sliding speeds, by as much as 17.3%–23.6% and 26.5%–32.6%, respectively, compared with the reference oil. This is related to the formation and deposition of a tribo-layer as a coating film on the frictional interfaces. Additionally, another important reason for the COF decline is the ability of Cu nanoparticles to convert sliding friction into rolling friction, which reduces the interaction between the worn surfaces. It is worth mentioning that the improvement in the anti-friction while using the Cu/Gr nano-lubricants is higher than that of the Cu nanolubricants, due to the synergistic impacts of the Cu/Gr hybrid nano-additive mechanisms.

As is well known, wear and friction do not occur on one material. Thus, the WR of the liner and ring is presented in the current results. The WR of the ring and liner results as a function of sliding distance for both the base oil and nano-additives under 307 N contact load and 0.39 m/s sliding speed are displayed in Fig. 7. The results demonstrate that the WR of the ring and liner samples for both base oil and nano-additives increased against the sliding distance owing to the lack of effectiveness of the rubbing surfaces in
maintaining the oxidized films as well as the change in wear mechanism the tribo-oxidation to adhesion. It is positively observed that Cu and Cu/Gr nano-additives decrease the WR of the ring and liner in the ranges of 13%–20% and 25%–30%, respectively, compared to base oil. This can be explained by the tribolayer deposition on the frictional surfaces, which suppresses the wear of the liner and ring owing to the self-replenishment of the tribofilms, as shown in Figs. 8 and 9, which increases the durability of the engine frictional surfaces.

3.3 Morphological analysis of the rubbing surfaces

To explain the wear mechanism of the liner at the TDC location, the rubbing surfaces of the cylinder liner when lubricated by the reference oil and nano-additives are exhibited in Fig. 8. As presented in Fig. 8(a), the plateau honing surface appeared on the rubbing surface before the sliding. As is well known, the plateau-honed liner surface confirms simultaneously
the sliding characteristics of a smooth surface and the excellent potentiality in maintaining lube oil on the rubbing surface. Moreover, the honed liner surface can provide high anti-wear during the friction process, although the rough surfaces assure high anti-seizure [34]. Figure 8(b) shows the evidence of scuffing (spalling pits, peeling) on adhesive junctions occurs when the liner surface is lubricated by the reference oil (5W-30). This is related to the breakthrough of the hard protrusions or asperities on piston ring surface into the worn surface of the liner-removing materials by plowing, which ultimately led to scuffing and increased WR and COF, as illustrated in Figs. 6 and 7.

In the lubrication of Cu nano-additives (Fig. 8(c)), the rubbing surface is covered with discontinuous tribo-layers of the Cu nanoparticles, as confirmed by the EDS and illustrated in Fig. 9. Hence, it has been noted that the anti-scuffing effect increased owing to the replenishment of the tribo-layers as a coating film. Meanwhile, the lubrication via the Cu/Gr nano lubricants prevents the abrasive and adhesive wear of the rubbing surfaces. As in Fig. 8(d), there is a tribofilm formed on the frictional surfaces that covers wide scratches, leading to a smoother worn surface and self-healing. Moreover, the synergetic effects of Cu and Gr present superior anti-wear characteristics, as revealed in Fig. 7. The frictional surfaces showed that the filling of the micro-asperities of the worn surfaces by nano-additives is the first mechanism. Sequentially, the thermal activation in the contact area is responsible for producing self-lubricating film via a tribochemical reaction and electrostatic adhesion for the iron debris particles and nano-additives on the substrate surface, as reported by Ali et al. [15]. As a result, the formation of the lubricating layer helps in delaying or preventing the occurrence of adhesive wear (scuffing damage),

![Tribo-lubricating layers formed on the cross section of the ring lubricated by: (a) reference oil, (b) Cu nanolubricant, (c) Cu/Gr nanolubricant, and (d, e) EDS mapping and the spectrum of tribofilm elements, corresponding to the yellow dash line box in (c).](image-url)
as presented in Fig. 8(d).

The nanostructure and elemental composition of the tribo-layers on the worn surface of the ring were examined using FESEM and EDS analysis on the cross section when lubricated by the reference oil, Cu, and Cu/Gr nanolubricants, as shown in Fig. 9. From the FESEM image in Fig. 9(a), the tribo-layer can be classified into a compacted layer owing to both the engine oil additives and wear debris. Furthermore, Figs. 9(b) and 9(c) show the tribo-layer morphology, containing Cu and Gr nano-additives, wear debris, and other organic compounds, which explain the uniform formation of the self-tribofilm on the frictional surface. Meanwhile, the tribo-layer can act as a coating layer to reduce the metal-to-metal contact in the rubbing surfaces. Figures 9(d) and 9(e) show the elemental distribution of EDS maps of C, Cu, O, P, Si, Ca, S, and Fe, respectively. Based on the EDS elemental characterizations, the compositions of the tribo-lubricating layers on the rubbing surfaces were Cu and C from nano-additives, wear debris from the substrate surface (Fe, O, and Si), and other compounds (P, S, and Ca) from a fully formulated engine oil (5W-30).

Figure 10 shows the 3D morphologies of the wear tracks of the liner supported by surface roughness profile for the reference base oil lubrication and Cu/Gr nanolubricants. The 3D results showed that the mean surface roughness values (Sa) of the liner lubricated by nano-additives decreased by 47.8% compared with that of the liner lubricated by the reference oil. This is due to the positive influences of the tribo-lubricating layers produced on the rubbing surfaces from nano-additives, which weakened the asperities tips, as presented in Figs. 8(d) and 9(c). Furthermore, the mean depth of the wear scar of the liner lubricated by the reference oil was 147.49 μm. Meanwhile, the liner lubricated by Cu/Gr nano-additives exhibited a wear scar mean depth of 72.38 μm, as shown in Fig. 10(b). These results indicate that the replenishment of self-lubricating films occurred on the worn liner surface when lubricated by nano-additives.

4 Conclusions

The tribological test results showed that the COF decreased in the ranges of 17.3%–23.6% and 26.5%–32.6% for the Cu and Cu/Gr nano-lubricants, respectively, as compared with the reference oil. The WR of the ring and liner was also reduced by 13%–20% and 25%–30% for the Cu and Cu/Gr nano-lubricants, respectively. Furthermore, the surface roughness of the liner lubricated with Cu/Gr nanolubricants declined by 47%, compared to that of the reference oil. The morphology of the frictional surfaces showed a severe adhesion due to the removal of tribofilms, such as oxides, tearing, breaking, and melting of metallic junctions during the lubrication by engine oil without nanomaterials. Meanwhile, the Cu and Gr nano-additive-based lubrication presents smooth worn surfaces because of the active role of the nano-additives in the formation of the protecting tribo-layers on the frictional surfaces of the ring and liner. Hence, an
increase in the durability of the ring and liner will result in, which can cause a reduction in the gas leakage from the combustion chamber to the crankcase, leading to increased compression pressure inside the cylinder. In light of these results, it can be stated that the energy produced from the engine will be increased with reduced emissions and enhanced fuel economy. In the future, further investigations should discuss the engine performance and exhaust emissions under lubrication by Cu/Gr nano-additives during various standard driving cycles, as compared with a commercial reference oil.

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