Investigation of the tribology behaviour of the graphene nanosheets as oil additives on textured alloy cast iron surface

Dan Zheng¹, Zhen-bing Cai^{1*}, Ming-xue Shen², Zheng-yang Li¹ and Min-hao Zhu¹

¹Tribology Research Institute, Key Lab of Advanced Technologies of Materials, Southwest Jiaotong University, Chengdu, 610031, China

²Engineering Research Center of Process Equipment and Its Remanufacture, Ministry of Education, Zhejiang University of Technology, Hangzhou 310032, China

* Corresponding author: 86-02887600601, czb-jiaoda@126.com

Abstract: Tribological properties of graphene nanosheets (GNS) as lubricating oil additives on textured surfaces were investigated using a UMT-2 tribotester. The lubricating fluids keeping a constant temperature of 100°C were applied to a GCr15 steel ball and an RTCr2 alloy cast iron plate with various texture designs (original surface, dimple density of 22.1%, 19.6% and 44.2%). The oil with GNS adding showed good tribological properties (wear reduced 50%), especially on the textured surfaces (the reduction in wear was high at over 90%). A combined effect between GNS additives and laser surface texturing (LST) was revealed, which is not a simple superposition of the two factors mentioned. A mechanism is proposed to explain for these results -the graphene layers sheared at the sliding contact interfaces, and form a protective film, which is closely related with the GNS structures and surface texture patterns

Keywords: graphene nanosheets, oil additives, laser surface texturing, wear reduction

1 Introduction

High efficiency, long durability and safety: an eternal theme of moving mechanical systems. However, it has been well known for some time now that a large number of our non-renewable energy resources were used to overcome friction, and a large number of mechanical system failure originated from wear [1]. Consequently, research and industrial application of tribology has been paid more and more attentions [2]. Diana et al. [3] believe that one of the most effective ways to control or reduction friction is to use a lubricant in liquid and/or solid forms. Recently, the study of carbon nanomaterials used as lubricant additives were widely reported. Alazemi [4] et al. found that ultrasmooth submicrometer carbon spheres as lubricant additives manifested good tribological properties for friction and wear reduction. Carbon nanotubes have been studied as lubricant additives by Yu et al., and shown good tribological performance [5,6].

Graphene, a single atom thick carbon with a 2D structure, was derived from a single layered sheet of graphite by Novoselov and Geim first time [7], and has attracted great attention due to its unique properties (including lubricating potential). Zhang et al. [8] found that oleic acid-modified graphene shown good tribological performance in lubricating oil. The study by Kinoshita et al. [9] showed that graphene oxide sheets function as an excellent water lubricant additive to reduce friction and surface wear. Mao et al. [10] have studied that graphene as a lubricant on Ag for electrical contact applications. Simultaneously, friction and wear properties of different types of graphene nanosheets have been studied as effective solid lubricant by Peng et al. [11]. All of these studies showed that graphene has a potential and promising tribological application. However, the research on pure graphene used as lubricating oil additives has rarely been reported because graphene is neither hydrophilic nor lipophilic [12].

In addition, surface texturing technique is another commonly used method in surface engineering. And its friction reducing and anti-wear mechanisms have been widely reported as a decrease in real contact area, wear debris entrapment and lubricating oil storing [13-16]. Recently, various surface texturing techniques, such as mechanical machining [17], chemical etching [18], and laser texturing [19,20] have been widely used to improve the tribological

characteristics of rubbing interface. Among these, laser surface texturing (LST) offers some obvious advantages, such as versatility, fast adaptability, high precision and cleanness of environment [19,21].

To date, most of the research on lubricant additives has been conducted on smooth surfaces. The tribological properties of lubricant additives on textured surfaces are worth studying and exploring. In this study, tribological properties of graphene nanosheets (GNS) as lubricating oil additives on textured surfaces were investigated on sliding steel-steel interfaces at a constant temperature of 100°C by using a UMT-2 ball-on-plate tribotester, and a combined effect of these two factors on reducing the friction and wear was revealed. The details were provided in the Supporting Information.

2 Materials and methods

2.1 Material processing and sample preparation

The graphene used in this study was purchased from Jinan Graphene New Materials Co., Ltd, and the trademark is MX-AVS. Its structural properties were measured by Atomic force microscopy (AFM, SPI3800N) and Raman spectroscopy (LabRam HR) equipped with a 532 nm laser.

The ball sample was GCr15 steel (wt %: 1.0 C, 1.49 Cr, 0.31 Mn, 0.26 Si, 0.009 P, 0.004 S) with a diameter of 9.525 mm and hardness of 766 HV. The plate sample was RTCr2 alloy cast iron (wt %, 3.11 C, 2.21 Si, 0.50 Mn, 1.63 Cr) with hardness of 220 HV, which was cut into 25 × 12 × 6 mm sheets and subsequently polished to obtain a surface roughness of $Ra \leq 0.03 \mu\text{m}$. Micro-dimple patterns were then created on the RTCr2 plate surfaces through an Nd: YAG laser with the wavelength of 1064 nm, the pulse frequency of 10 kHz, the average power of 10 W and the scanning speed of 5 mm/s. After laser texturing, all samples were re-polished gently to remove bulges or burrs around the rim of the dimples and obtain a roughness of $Ra \leq 0.03 \mu\text{m}$. Three dimple densities with the same dimple geometric parameters were designed. A smooth surface was also used to provide a baseline for comparison. These four plate samples were denoted as 0#, 1#, 2# and 3# respectively. The details of the parameters are shown in Table 1.

Table 1 Characteristics of various laser textures

Samples	0#	1#	2#	3#
Pattern	Original
Dimple density (%)	-	22.1	19.6	44.2
Dimple spacing (X, μm)	-	400	300	200
Dimple diameter (μm)	-	150	150	150
Dimple depth (μm)	-	30	30	30

2.2 Experimental procedure

Tribological tests were performed using a UMT-2 tribometer in the ball-on-plate reciprocating wear mode with applied load of 10 N, sliding distance of 8 mm, speed of 5 mm/s, and the sliding direction parallel to the LST patterns. The upper ball sample and the lower plate sample were fixed in a square tank equipped with lubricating oil. The bottom of the tank was fitted with a heating device that can control the experimental temperature. Before each test, the balls and plates were cleaned with ethanol to keep the surfaces as clean as possible. In this study, 0.01 wt % GNS was added into PAO4 oil as lubricant. Considering the difficulty in preparing stable dispersion of graphene [22-24], 1 wt % Span-80 ($\text{C}_{24}\text{H}_{44}\text{O}_6$, Chengdu, Changzheng Glass Co., Ltd.) was used as a dispersing agent, and the mixed oil was stirred for over 10 min and then subjected to 15 min of ultrasonic to create a uniform suspension in oil. PAO4 base oil was also used to provide a baseline for comparison. Two series of tribological tests, including those under PAO4 base oil lubrication and GNS dispersed oil lubrication conditions, were run for 6000s at a constant temperature of 100°C with the same test parameters.

After the tests, the ball and plate samples were cleansed with ethanol. The morphologies and tribochemistry of worn surfaces were analysed through 2D profiler (NanoMap-D), optical microscopy (OM, OLYMPUS BX60M), scanning electron microscopy (SEM, JSM-7001F), energy dispersive X-ray spectroscopy (EDX, EDAX-7760/68M) and Raman spectroscopy

(LabRam HR). To estimate the wear rate after the tests, we calculated the wear volume of the plate side as follow.

3 Results and discussion

3.1 Characterization of GNS and texture

Fig. 1a shows the AFM image of the GNS sample. The sample was prepared by dropping a 0.01 wt % GNS dispersion onto a freshly cleaved Si substrate followed by drying in air. The height of the GNS sample was measured at around 1.5 nm. It concluded that the GNS had a few-layer structure. To obtain additional information on the GNS structure, Raman spectrum of the GNS sample was conducted and shown in Fig. 1b. It seen that the intensity of G peak is higher than that of 2D peak, and the 2D peak of the GNS sample can be fitted with six Lorenz peaks. It is likely that the GNS was three layers [25-27]. In summary, the GNS used in this study had a few-layer structure.

Fig. 2 shows the digital pictures of GNS dispersions with and without addition of 1 wt % Span-80 immediately after sonication (a) and 4 weeks after sonication (b). 0.05 wt % GNS solution was selected for better observation. After adding Span-80, the GNS additives were well dispersed in PAO4 oil, probably due to the edge sites and basal planes of GNS was coated with the surfactant Span-80, which made GNS partially oleophilic that guarantees the stable existence of GNS in PAO4 oil. However, the GNS dispersion without addition of Span-80 displayed only short-term stability and precipitated completely in a matter of hours to a few days. By contrast, it shown that the surfactant Span-80 effectively allows GNS to disperse well in PAO4 oil.

3D morphologies of plate samples with various surface textures are shown in Fig. 3. It can be seen that the micro-dimples are arranged regularly on the surfaces.

Fig. 1 Characterization of the GNS.

Fig. 2 Digital pictures of GNS dispersions with and without addition of 1 wt % Span-80 immediately after sonication (a) and 4 weeks after sonication (b).

Fig. 3 3D morphologies of plate samples. (a) 0#, (b) 1#, (c) 2# and (d) 3#.

3.2 Friction and wear

Fig. 4 shows the curves of the friction coefficient (COFs) of various textured surfaces under (a) PAO4 base oil and (b) GNS dispersed oil lubrication. For the condition of PAO4 base oil lubrication, the COF presented relatively high values and acute fluctuation (Fig. 4a). And surface textures resulted in higher COF, probably due to the increase in surface roughness leading to greater resistance at the sliding contact interfaces [28], implying that surface textures have effect on enhancing friction. Furthermore, the COF of 3# was significantly lower than that of 1# and 2#, probably due to the lubricating oil and wear debris storing of micro-dimples [29]. As shown in Fig. 4b, friction was effectively reduced and more stable after adding GNS additives. For example, the COF of 0# was reduced to approximately 0.29 from approximately 0.59. This COF reduction is thought to be attributed to the combined lubrication effect of Span-80 and GNS additives. However, tests performed with Span-80 dispersed oil (PAO4 base oil + 1 wt % Span-80) lubrication showed a same COF as that of PAO4 base oil lubrication (not shown here), suggesting that the GNS is most likely the reason for the friction reduction.

In the following, the impact on wear of surface textures and GNS additives will be discussed. Considering the wear on the balls was too low to quantify, worn plate scars were mainly analysed in this study. The wear rates of plates are compared in Fig. 5; cross-sectional profiles of plate wear scars are also shown in Fig. 5. As shown, the wear rate of the original friction and lubrication condition (both surface textures and GNS additives were not used) presented a high value. However, relatively higher values of the wear rates were presented when surface texturing was used; indicating that surface texturing not only increases the friction but also increases the wear. As was expected, the wear rate was effectively reduced with the addition of GNS, for instance, the wear rate of 0# reduced from 778 to 371. It cannot be overlooked that the wear rates of textured plates were significantly lower than that of the original one for the tests performed in GNS dispersed oil. This finding clearly reveals the combined effect on reducing wear between GNS and surface textures, and it is not a simple superposition of the two factors mentioned.

Fig. 4 Friction coefficient curves of various textured surfaces under (a) PAO4 base oil and (b) GNS dispersed oil lubrication.

Fig. 5 Comparison of wear of plate samples with various surface textures. (a) Wear rates of the worn plates and cross-sectional profiles of plate wear scars under (b) PAO4 base oil and (c) GNS dispersed oil lubrication.

3.3 Morphological analysis of worn surfaces

The optical morphologies of the worn surfaces of the balls and various textured plates tested in PAO4 oil with and without addition of GNS are presented in Fig. 6. The wear scars of balls present oval shape instead of circular shape, and the explanation have been proffered by Cai et al. [30]: the contact area of ball and plate in the sliding direction is smaller than those in the vertical direction, because the plate material is worn off in the sliding direction. For wear scars generated under GNS dispersed oil lubrication, worn single dimple can be observed on the worn ball surfaces (zones surrounded by red circles), probably due to the local high pressure on the edge of micro-dimples. You can imagine that same phenomenon may occurred in the tests performed in PAO4 base oil but cannot observed because of wearing heavily. It's not difficult to understand that micro-dimples may lead to greater contact pressure at the sliding contact interface, and it may be the reason for the increase of friction and wear resulted from surface texturing. The width of plate wear scars is also indicated in Fig. 6.

Samples	PAO4		PAO4+GNS	
	Ball	Plate	Ball	Plate
0#
1#
2#
3#

Fig. 6 Optical morphologies of worn surfaces.

The wear morphologies of various textured plates tested in PAO4 oil with and without addition of GNS were investigated by SEM. As can be seen in Fig. 7a,b, the worn scar of o#

under PAO4 base oil lubrication displays deep machining marks and the material spall seriously, indicating that severe scuffing occurred in this case. After adding GNS additives, the defects become relatively shallow and narrow and the worn surface becomes smoother, despite the presence of machining marks (Fig. 7c,d), showing that the GNS may offer protection to the sliding interface. Fig. 8 to Fig. 10 display the combined effect between surface texturing and GNS additives on reducing wear. When both texture and additive were used, the worn surfaces were significantly smoother and the defects were not obvious comparing with that under single effect of GNS additives. In addition, the worn surface of 2# under GNS dispersed oil lubrication shows relatively complete residue micro-dimples and slight defects than that of 1#, suggesting that 2# have better tribological property than 1# despite the similar dimple density, probably due to the smaller dimple spacing in the sliding direction of 2#. The best anti-wear performance appeared on the condition of GNS-3#, and the most probable cause is that micro-dimples can store the GNS additives.

Fig. 7 SEM morphologies recorded at different magnifications of worn plate surfaces of 0# tested in (a-b) PAO4 base oil and (c-d) GNS dispersed oil.

Fig. 8 SEM morphologies recorded at different magnifications of worn plate surfaces of 1# tested in (a-b) PAO4 base oil and (c-d) GNS dispersed oil.

Fig. 9 SEM morphologies recorded at different magnifications of worn plate surfaces of 2# tested in (a-b) PAO4 base oil and (c-d) GNS dispersed oil.

Fig. 10 SEM morphologies recorded at different magnifications of worn plate surfaces of 3# tested in (a-b) PAO4 base oil and (c-d) GNS dispersed oil.

3.4 EDX and Raman analysis of friction contact regions

To expound the wear reduction mechanisms in depth, local structures were characterized using energy-dispersive x-ray spectroscopy (EDX) and Raman spectroscopy. For 3#, the EDX spectra obtained from the worn and un-contact area under GNS dispersed oil lubrication are shown in Fig. 11. Table 2 shows the elemental content corresponding to the EDX spectra in Fig. 11. It is obvious that O element appears on the worn area, while only C and Fe exist on the un-contact area. And the C content of the worn area is significantly higher than that of the un-contact area. In addition, it the highest when the analysis location was position 1 of the worn dimple (it increased to 53.17% from 31.49% compared with that in position 2). Thus, it inferred that the tribological properties of GNS may be ascribed to a protective film caused by adsorption of GNS to the surfaces of the sliding parts. Another evidence was provided by Raman measurements as fellow.

Fig. 11 EDX spectra obtained from different locations on the worn plate of 3#. The right image indicates the approximate positions where EDX spectra were obtained.

Table 2 Elemental content corresponding to the EDX spectra in Fig. 11

Positions	C (%)	O (%)	Fe (%)
1	53.17	13.32	33.51
2	31.49	16.20	52.31
3	20.16	0	79.84
4	9.76	0	90.24

Fig. 12 shows the Raman spectra of different plate samples inside the wear scars and avoid the residual dimples under GNS dispersed oil lubrication. For all case, disordered GNS adsorbed on the sliding interfaces during the tribotests, as evidenced from the disappearance of 2D peak in the wear scars, which is consistent with the findings of EDX measurements. At the same time, the D and G peaks were observed, representing the typical characteristic of GNS additives. To compare the Raman spectra of different samples, Gaussian fitting was applied for the D and G peaks fitting (Fig. 13(a-d)). The fitting results of these two peaks as well as the intensity ratio of the D peak to G peak (I_D/I_G) are also shown in Fig. 13. As shown in Fig. 13(f-g), not only the intensity of the D and G peaks but also the I_D/I_G follows the order: $0\# < 1\# < 2\# < 3\#$. In the Raman spectra of graphene, the I_D/I_G can be used to characterize the sp^3/sp^2 bonding ratio [31]. As indicated by Ferrari and Robertson's three-stage model for the Raman spectra of disordered and amorphous carbon [32], GNS additives on the textured surfaces underwent more significant graphitization than that on the original one during

friction, probably attributed to the large amount of graphitized wear debris entrapped in the micro-dimples. The condition of GNS-3# exhibited the most obvious graphitizing transformation after the friction testing, considering its largest dimple density. However, the I_D/I_G of GNS-1# condition was notably lower than that of GNS-2# condition despite the similar dimple density, which is unexpected, but consistent with the results of the wear rates, probably due to the larger dimple spacing in the sliding direction of 1#.

These results indicated that GNS displays excellent properties on reducing friction and wear as additives in PAO4 oil, especially evident on the textured surfaces. The low friction and wear reduction may be due to the 2D structure and extreme strength of graphene. Fig. 14 illustrates the effects of surface texturing and GNS additives on the friction and wear behaviour. In this study, a slow sliding speed of 5 mm/s and a relatively high temperature of 100°C were employed and hence correspond to a boundary lubrication situation. The PAO4 base oil can be easily cut off due to the high contact stress achieved from the small contact zone under the ball-on-plate contact model on the untextured plate (Fig. 14a). Additionally, comparing with the original plate, the textured plates exhibit relatively high friction and wear increase in PAO4 base oil. A possible explanation is that the boundary lubricating film rupture caused by the local high stress on the dimple edge and, hence, provides high friction (Fig. 14b). After GNS adding, the graphene layers sheared at the sliding contact interfaces attributed to its 2D structure [33, 34], and the oil film strength was improved (Fig. 14c). On the textured surfaces, the micro-dimples can store the GNS additives and release and, hence, improves the contact state of the friction interfaces (Fig. 14d). The GNS additives adsorbed on the sliding interfaces, and a protective film may be formed as confirmed by the EDX and Raman measurements (Fig. 11-13). Furthermore, the micro-dimples resulted in high contact stress on the sliding interfaces. However, it is also responsible for graphitization of GNS additives and, hence, promotes the adsorption of GNS. It is a possible explanation for the significant combined effect of GNS additives and surface texturing on reducing friction and wear, which is closely related with GNS structure and texture patterns.

Fig. 12 Raman spectra of different plate samples inside the wear scar and avoid the residual dimple under GNS dispersed oil lubrication.

4 Conclusions

The combined effect of GNS as lubricating oil additives and laser surface texturing on reducing the friction and wear was investigated. High friction and wear increase was achieved under laser surface texturing alone affected because the high contact stress caused by the micro-dimples. At the same time, micro-dimples can store lubricating oil and trap wear debris, hence, reduce friction and wear. The GNS additives modified by Span-80 were well dispersed in PAO4 oil, and showed a good tribological properties (wear rate was reduced approximately 50% after adding GNS), especially on the textured surfaces (the reduction in wear was high at over 90%). Condition of GNS-3# showed the optimal tribological properties. These findings can be explained by the combined effect between micro-dimples and GNS additives: induce graphitizing transformation and the storing of GNS additives of the micro-dimples. It was confirmed by the characterization performed after tribotests, and indicating that the graphene layers sheared at the sliding contact interfaces, and a protective film was formed, which exhibited higher strength on the textured surfaces due to GNS storing and accelerating effect on the graphitization of GNS additives of micro-dimples. Furthermore, GNS structure and texture patterns are important factors influencing friction and wear properties.

Fig. 13 Raman analysis. (a-d) Gaussian fitting applied for D and G peaks fitting of 0#, 1#, 2# and 3# respectively. (e-f) The fitting results of the D and G peaks as well as the intensity ratio of the D peak to G peak (I_D/I_G).

Fig. 14 Schematic diagram of the wear mechanism.

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