

## EFFECT OF ECO-FRIENDLY CARBON MICROSPHERES ON THE TRIBOLOGICAL BEHAVIOR OF LITHIUM LUBRICATING GREASE

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*Eco-friendly carbon spheres (CS) used for lithium grease were synthesized by hydrothermal synthesis in this work. The obtained composite greases were evaluated on a HSR-2M ball-on-disk friction machine to explore the frictional and wear behaviors under different additive contents and loads. The morphology of worn surface is analyzed by scanning electron microscope (SEM) and three-dimensional surface profiler. Results showed that when the content of CS in grease is 1wt% at the load of 60N, the friction coefficient (COF) is reduced by more than 20% compared with that of base grease. The worn surface is quite smooth with few shadow furrows and the wear volume decrease by more than 40% compare with base grease when the content of CS is 1wt%. The results indicated that CS displayed excellent anti-wear ability and friction reducing performance as lubricating grease additive. The lubricating mechanisms of CS were the 'micro-roller' effects, repairing and forming the protective film.*

**Keywords:** carbon microspheres; lithium grease; tribological properties; worn surface

### 1. Introduction

The vast majority of rolling bearings use greases as lubricants, which plays an important role in maintaining the normal operation of all kinds of mechanical equipment, reducing the friction and wear, prolonging the service life of mechanical equipment [1,2]. However, as we know, the performance of grease lubrication depends largely on the properties of the additives [3,4]. Therefore, the research on lubricating grease additives has become a key factor to determine the development of grease and developing efficient lubrication additives will bring

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economic and social benefits greatly [5,6]. Therefore, the research of new lubricant additives has been a hot research topic all over the world [7,8].

An increasing number of literatures emphasize the importance of using micro and nanoparticles as lubrication additives due to their likelihood to improve the tribological performance of lubricant [9,10]. Research shows that adding some kinds of micro and nanoparticles in lubricating oil can form a stable suspension and exhibit excellent anti-wear and antifriction properties of extreme pressure [11-13]. Recently, Jorge et al. reported the addition of the long-chain capped  $ZrO_2$  NPs in base lubricating oils exhibited low friction coefficients and improved the anti-wear properties of the base oil when compared with the raw lubricating oil [14]. Pol. demonstrated that adding 1.0wt% of carbon spheres reduced both friction and wear under boundary-lubricated sliding conditions [15]. In addition, Alazemi et al. introduced carbon spheres as an efficient additive in lubricating oils exhibited a substantial reduction in friction and wear (10-25%) compared to the neat oil, without change in the viscosity [16].

In recent years, development in the field of nanotechnology has led to the growth of nanoparticles as lubricant additives. In the research of lubricating grease additives, it has been also developed to a new level [17-19]. Ji et al. synthesized nanometer  $CaCO_3$  via the carbonation method as additives in lithium grease and found that these  $CaCO_3$  nanoparticles exhibit good performance in anti-wear and friction-reduction, load-carrying capacity and extreme pressure properties [20]. Elshalakny et al. stated that the multilayer nanometer graphene platelets(MLNGPs)/multiwall carbon nanotubes(MWCNTs) as an additive on lithium grease, and the results showed that the WSD was reduced by 66%, COF was reduced by 91%, while maximum nonseizer load increased by 90 kg over ordinary lithium grease when the concentration of MLNGPs is 1% [21]. Wang et al. studied the tribological properties of the grease with GN/Cu NPs composites and found that the GN/Cu NPs composites could help to reduce the wear loss of the disk by 85.5% and the average friction coefficient by 15.5% compared to the base grease [22]. Most researches in the field of grease additives have only focused on nanometer particles or other carbon materials like carbon nanotube, graphene, etc.

To the best of our knowledge, no study has investigated that CS is used as an effective grease-lubricating additive. In this paper, eco-friendly carbon spheres (CSs) with the size from 600nm to  $1\mu m$  were prepared by hydrothermal synthesis; the spheres were observed and evaluated by Field-emission scanning electron microscope (FESEM). CSs were combined into the base lithium grease, can improve the tribological properties of base lithium grease, and anti-wear and friction-reducing performance on the anti-wear tester was studied. The friction coefficient, wear volume and worn morphology of base lithium greases with different carbon microspheres were discussed and analyzed. The main aim of this

study is to investigate the performance of CS as a lubricating additive and its mechanism. At the same time, we hope to prepare effective lubrication additive to reduce the friction and wear of the machine so as to save energy.

## 2. Experimental

### 2.1. Preparation of the CSs

3# lithium base grease (provided by Weixian special oil chemical plant in China) was used as base grease. CSs were prepared by the reported one step hydrothermal carbonization of glucose. The reason why glucose is chosen as the carbon source is that compared with other carbon sources, such as sugar, sucrose and  $\beta$  -cyclodextrin, the hydrothermal reaction products of monosaccharide glucose are relatively pure with only amorphous carbon, while the products of disaccharides and polysaccharides contain aromatic hydrocarbons [23]. In a typical experiment, 12.6g glucose was dissolved in 70mL distilled water under magnetic stirring. Then, the above solution was transferred into a stainless steel autoclave to be maintained at 180 °C for 10 h. After the product was cooled to room temperature, the CSs were obtained via centrifugation and washing with distilled water and ethanol several times [24].

### 2.2. Preparation of the modified grease

The carbon spheres with different contents were added to the base grease to form several lubricating greases with mass fraction of 0wt%, 0.0625wt%, 0.125wt%, 0.25wt%, 0.5wt%, 1wt% and 2wt%. It was marked as a, b, c, d, e, f, and g, respectively. The preparation method was as follows. 30g base grease was weighed in the container, and the CSs were added to the base grease to stir evenly. The stirred grease was grinded for 10min with the three roll grinder, so that the CSs were evenly distributed in the grease. At last, the modified grease was made.

### 2.3. Tribological tests

The tribological properties of the lithium grease containing CSs were evaluated using an HSR-2M ball-on-disk testing machine (Lanzhou science and technology development Co. Ltd, China) at room temperature. The balls and disks used in the tests were Gr15 stainless steel and the ball with a diameter of 5.953mm. The VB program on computer will calculate the sliding friction COF pairs on the basis of the average load value and the average force of friction, and show the curve of friction coefficient changing with time in real time to the program interface. Before the test, both steel disks and steel balls were cleaned by ethanol to remove impurities. The friction and wear tests were conducted under the load of 50N, 60N, and 70N. Test results were compared with the pure lithium

grease and a, b, c, d, e, f, g, respectively. The width and depth of the wear area were acquired from the 3D surface profiler.

#### 2.4. Microstructure tests

In order to further study the tribological mechanism of CS as a lubricating additive, high resolution scanning electron microscope (HRSEM, JSM-7800; Japan Electron Optics Laboratory Co. Ltd.) and desktop scanning electron microscope (EM-30; South Korea COXEM Company) were used to observe and evaluate the microscope morphology of CSs and the worn surface. The 3D surface morphology of the worn surface was investigated with a confocal microscope surface profiler (Vsurf Explorer, Nanofocus Inc.)

### 3. Results and discussion

#### 3.1. Structural properties of CSs

Studies have shown that the synthesis of carbon microspheres by hydrothermal method requires the polymerization of glues molecules, hydroxyl dehydration and the final carbonization process. When the size of the carbon sphere is very small (e.g., C<sub>60</sub> molecule is 1nm), it cannot play the role of rolling friction, but may be trapped in rough grooves on the surface, which is not conducive to the shear process. In this experiment, the size of the carbon sphere >600nm was prepared to make it larger than the roughness of the shear surface, so as to play the role of rolling friction [25]. Fig. 1 shows the morphology of CS investigated by FESEM. It is clear that the surface of the CS is smooth and perfectly spherical in size of 600nm-1μm and a slight agglomeration appears between the CSs. Fig. 2 presents the X-ray diffraction (XRD) of CS.

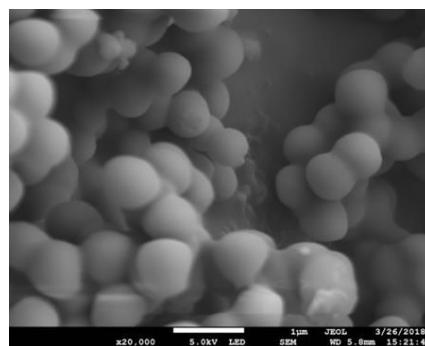


Fig. 1. SEM image of CS (SEM: scanning electron microscopy CS: carbon spheres)

The mild and broad peak at around 10~30° suggests a low degree of graphitization and possibly the presence of amorphous carbon and therefore do not easy to expose sharp surface facets such as crystals, which may damage the shear surface

and prevent effective rolling. Therefore, the existence of amorphous carbon is conducive to reducing the friction coefficient.

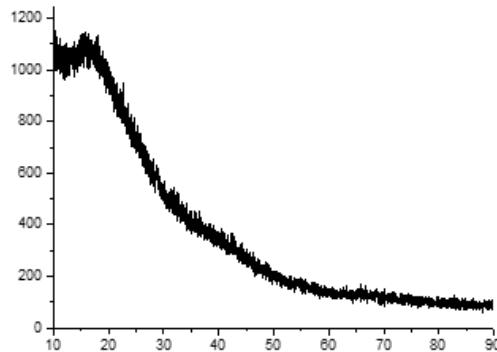


Fig. 2. XRD patterns of CMs

### 3.2. Tribological properties of modified greases

#### 3.2.1. Influence of concentration

Fig. 3 shows the average COF of lithium grease at different additive content: 0wt%, 0.125wt%, 0.25wt%, 0.5wt%, 1.0wt%, and 2.0wt%. All the experiments were carried out at a certain condition: 60N, 1HZ and 30min. It is demonstrated that all the COF of lithium grease filled with CS are lower than that of the base grease. With increasing the content of CS, mean COF decreases

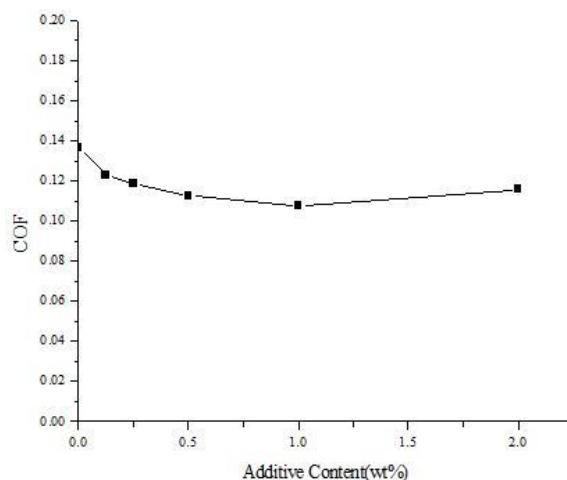


Fig. 3. The average COF of the grease at different additive contents.

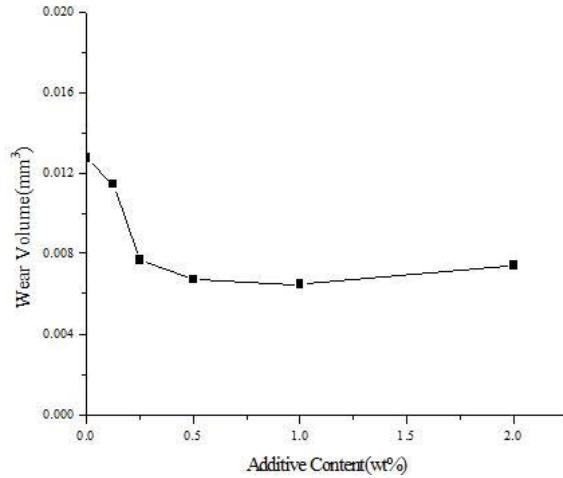


Fig. 4. The wear volume of the tribo-pair at different additive contents.

Gradually; when the content of CS is more than 1wt%, the mean COF begins to Increase. The average COF of lithium grease reaches the lowest value of 0.1076 at the CS content of 1.0wt%, reduced by 21.35% as compared with (0.1368) base lithium grease. It indicates evidently that the lithium base grease with proper amount of CS has better tribological performance. Fig. 4 demonstrates the wear volume of lithium grease with various content of CS. The trend of change about wear volume is similar to that of the mean COF. When the content of CS increases from 0 to 1.0wt%, the wear volume reduces from 0.012734mm<sup>3</sup>to 0.006456mm<sup>3</sup>, and the reduction rate is 49.28%. However, the wear volume arises when the content of CS surpasses 1.0wt%. Compared with these results, the modified grease shows the optimal anti-wear and anti-friction performance when the content of CS is 1wt%

### 3.2.2. Influence of load

Fig. 5 shows the average COF and wear volume of base grease and the modified grease containing 1.0wt% of CS at various loads. The mean COF of the base grease is higher than that of the modified at the same load, and it shows an increasing tendency as the loads increase. The average COF of modified grease at a load of 60N is a minimum, which decreases by 21.35% as compared with the COF of base grease. Therefore, it is optimal for the friction test under the condition of 60N when the content of CS is 1.0wt%. The wear volume of base grease at the load of 60N is 0.01273mm<sup>3</sup> while that of modified grease contained 1.0wt% CS decreases to 0.006456 mm<sup>3</sup>. It indicates that the modified grease containing 1.0wt% CS performs a better anti-wear at the load of 60N, which reduced by 49.29%. Accordingly, it has the same change trend with that of the

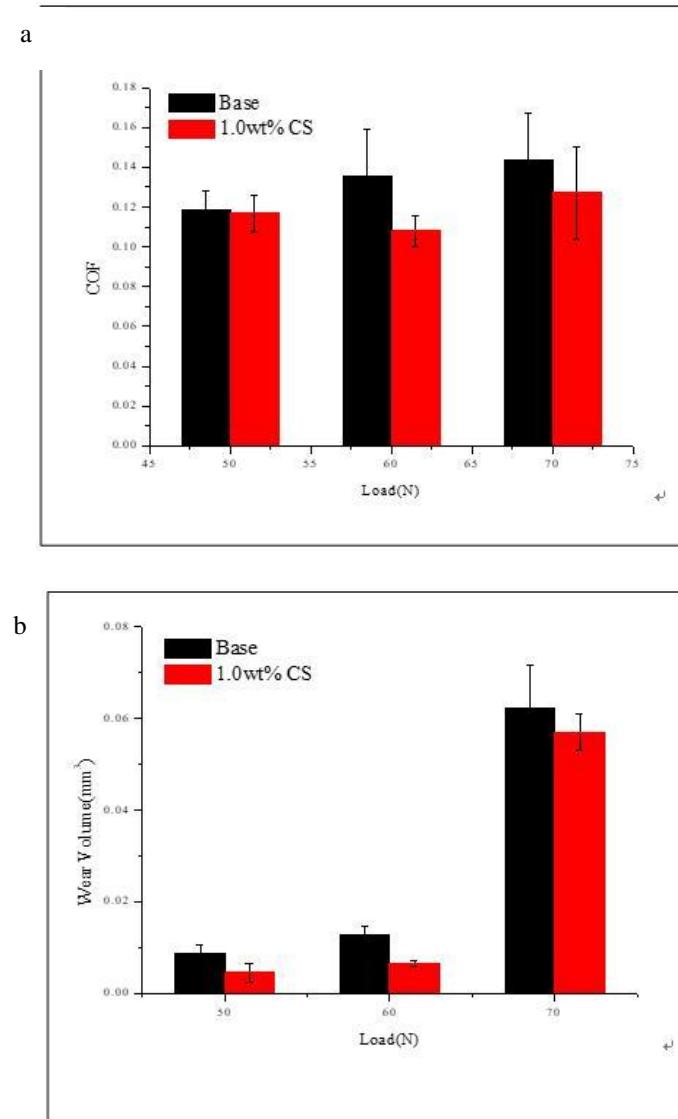


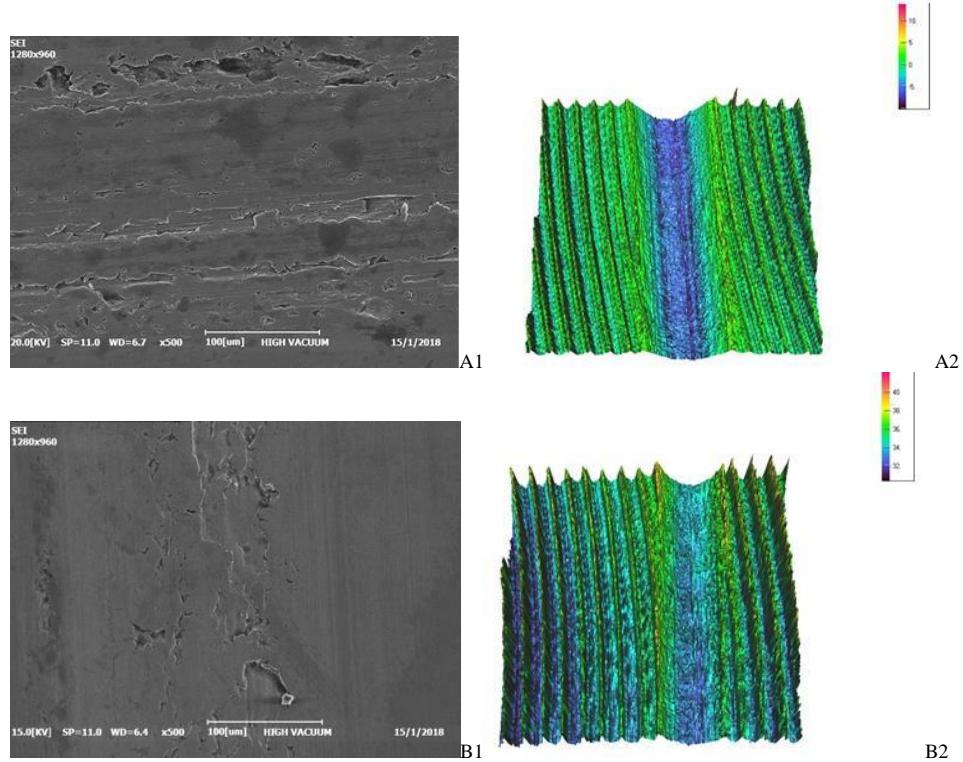
Fig. 5. Average COF (a) and WV (b) of base grease and modified grease.

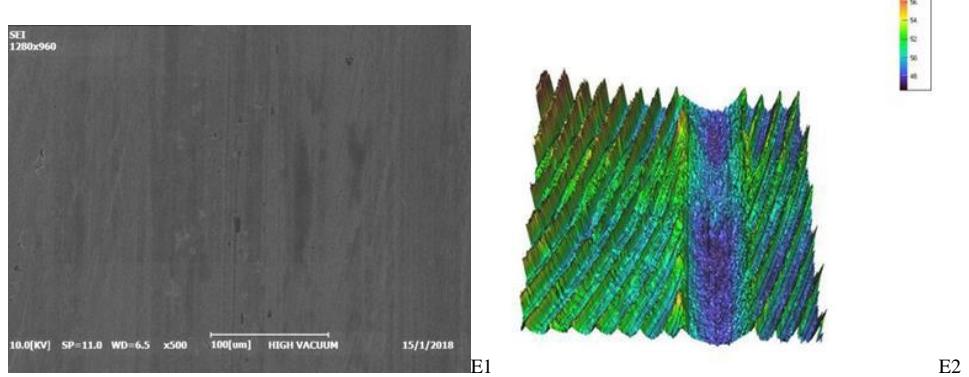
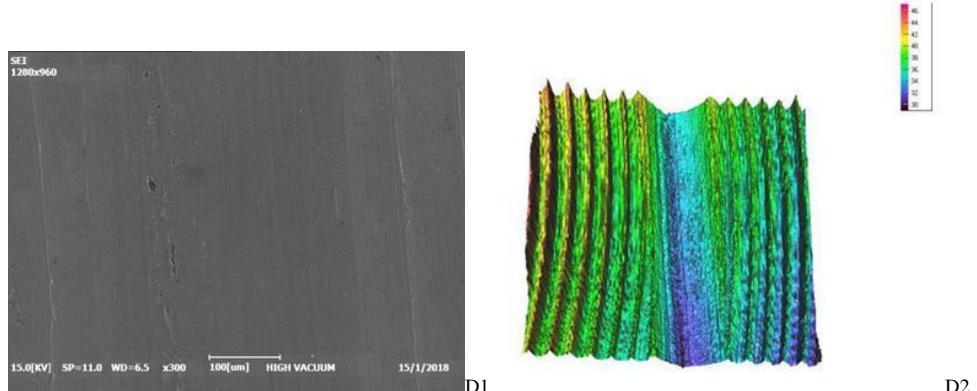
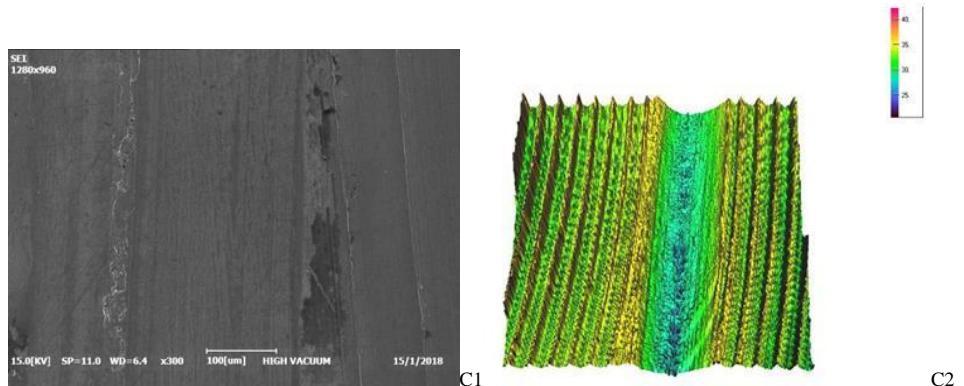
COF, which illustrates that the CS can improve the ability of anti-wear and anti-friction about lithium grease at the load of 60N

### 3.3. Analysis of worn surface

In order to study the tribological mechanism of carbon microspheres as lubricating additive on the sheet steel further, we used the three-dimensional surface profiler to characterize the 3D and 2D contour map of the surface and the corresponding grinding depth. In addition, the desktop scanning electron microscope was used to represent the morphology of the steel sheet surface. The

result is shown in Fig. 6. It is obvious that the worn surface of the steel disk lubricated by lithium grease alone is quite rough and shows wide and deep furrows as well as grooves along the sliding direction. Especially a large number of plastic deformation and brittle rupture occurred at the edge of the abrasion, as shown in A1 and A2 (Fig 6). When the content of CSs is 0.0625wt%, the improvement on the worn surface is not obvious. With the further increasing carbon microspheres, grinding crack width and depth reduce; it can be observed that the grinding mark is the narrowest and shallowest when the content of the CS is at 1%. The worn surface is most flat without debris and grooves, as shown in E1 and E2 (Fig 6), which well corresponds to the good anti-friction ability of the CSs. In contrast, when the content of CSs is more than 1wt%, the worn surface begins to become rough again; the friction coefficient increases as shown in G1 (Fig 6). The above-mentioned results illustrate that when the content of CSs is too low it cannot improve lubrication mainly due to a small amount of CSs having no chance to lubricate fully the contact point between steel disk and steel ball during friction. However, if the content of CSs is too much, the friction performance will reduce mainly on account of the big size of the agglomerated CS increasing the roughness of the contact surface. It is concluded that only proper content of CSs can make CS an effective additive in lithium grease.





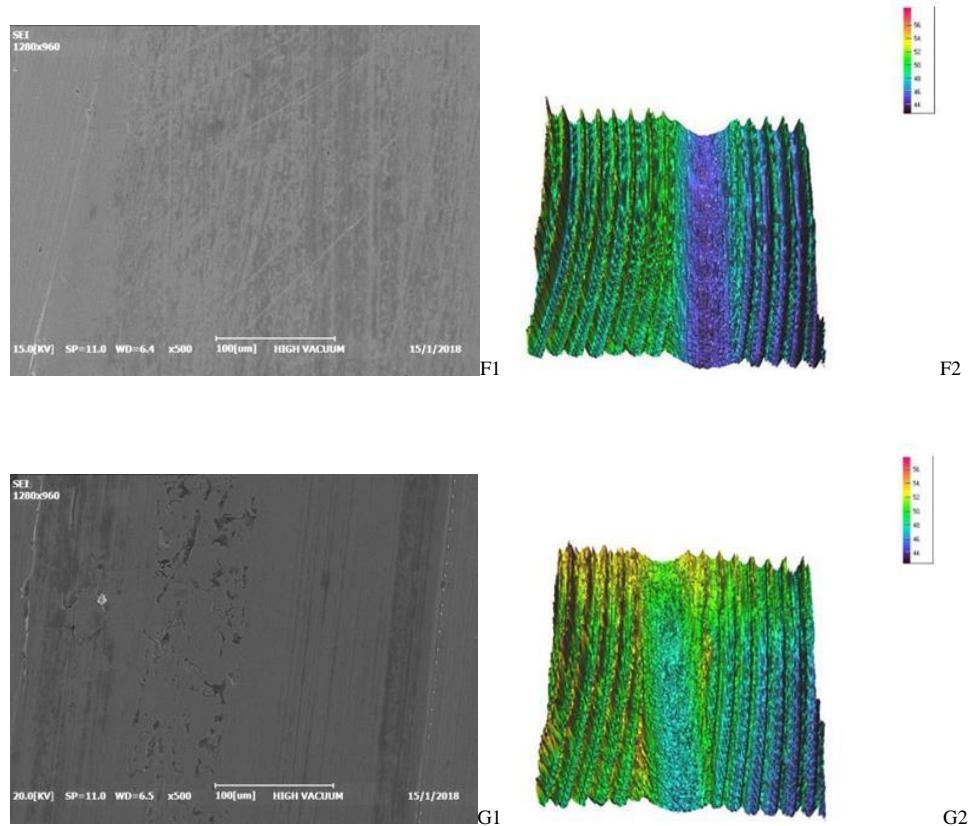


Fig. 6. SEM micrographs and three-dimensional morphologies of worn surface lubricated with different grease samples: A:base grease; B:grease containing 0.0625wt% CS; C:grease containing 0.125wt% CS; D:grease containing 0.25wt% CS; E:grease containing 0.5wt% CS; F:grease containing 1wt% CS; G:grease containing 2wt% CS;SEM: scanning electron microscopy; CS: carbon sphere

### 3.4 Anti-friction mechanism of carbon microsphere as lubricate additive

Based on the lubrication mechanism of other spherical nanometer or micron materials [24], we speculate that the carbon microspheres have the following role in antifriction and anti-wear mechanism combined with the structure characteristics of the carbon microsphere itself, as shown in Fig 7.

First, carbon microspheres have ‘micro-roller’ effects when they enter the friction surface. The ‘micro-roller’ effects refer to the fact that the particles can play a supporting role on the surface of the friction pair, so that the friction pair can convert sliding friction into rolling friction during the friction process, and the COF is reduced. The second aspect is that some carbon microspheres have very small particle sizes and it is easy to enter into the furrow in the process of friction,

where the carbon microsphere plays a filling and repairing role under the action of loads.

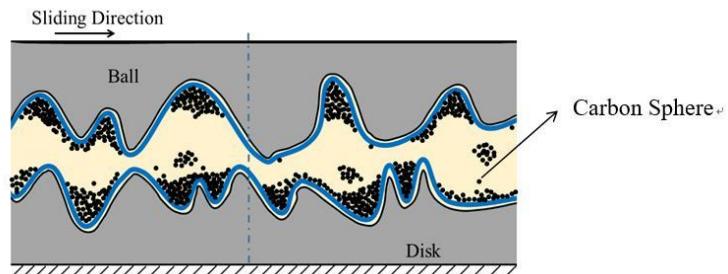


Fig. 7. Schematic diagram of tribological mechanism of carbon microspheres

Finally, the thickness of the grease becomes lower, the lubrication film becomes thinner and the contact points of friction increase under high temperature. At this point, the surface energy of carbon microspheres can easily react with the friction surface to produce the friction chemical protective film; thus, it can reduce the friction between the layer and the layer between the friction pairs. Therefore, the lubricating mechanism of the carbon microsphere lubricating additive in the grease is mainly the 'micro-roller' effects, filling and repairing, and forming the protective film.

#### 4. Conclusion

The anti-wear and anti-friction properties of CS prepared by hydrothermal method in lithium lubricating greases were studied on a reciprocating friction machine. The results showed that the optimum content and the optimum load were available. When the additive content of CS was 1.0wt%, the grease performed the most optimal anti-wear and anti-friction properties; COF was reduced by 20.43% and wear volume was reduced by 49.30% as compared with the base grease at the load of 60N. The discussion of lubricating role of CS showed that the mechanisms of CS were the 'micro-roller' effects, repairing and forming the protective film. Consequently, besides as the additive of lithium grease, the CS can be available to improve the tribological properties of the other grease.

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