

Article

Not peer-reviewed version

Study on the Influence of MoS₂ Addition Method on the Tribological and Corrosion Properties of Greases

[Can Zhu](#) , [Yi Zhong He](#) ^{*} , Ping li Xiong , [JiuSheng Li](#) , Li li Li

Posted Date: 16 November 2023

doi: 10.20944/preprints202311.1066.v1

Keywords: MoS₂; Lithium base grease; addition method; Friction; corro



Preprints.org is a free multidiscipline platform providing preprint service that is dedicated to making early versions of research outputs permanently available and citable. Preprints posted at Preprints.org appear in Web of Science, Crossref, Google Scholar, Scilit, Europe PMC.

Copyright: This is an open access article distributed under the Creative Commons Attribution License which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

Article

Study on the Influence of MoS₂ Addition Method on the Tribological and Corrosion Properties of Greases

Can Zhu ¹, Zhongyi He ^{1,*}, Liping Xiong ¹, Jiusheng Li ² and Lili Li ¹

¹ School of Materials Science and Engineering, East China Jiaotong University, Nanchang 330000, China

² Laboratory of Advanced Lubricating Materials, Shanghai Advanced Research Institute, Chinese Academy of Sciences, Shanghai 201210, China

* Correspondence: hzy220567@163.com

Abstract: MoS₂ lithium-based grease is suitable for lubrication protection between bearings at high temperatures and loads due to its excellent tribological properties. However, there is little research on the influence of different addition methods of MoS₂ additive on the tribology and corrosion properties of lithium grease. In this work, vegetable oil with low toxicity, high biodegradation rate, and low cost was selected as the base oil, lithium 12-hydroxystearate as the thickener, and MoS₂ as the additive. The effects of different adding modes of MoS₂ on the tribology and corrosion properties of lithium grease were studied. The experimental results showed that adding 0.01wt% MoS₂ before thickening was more conducive to improving the tribological properties of lithium grease. The average friction coefficient was 0.034, and the average spot diameter was reduced by 0.16mm. After grinding and adding 0.01wt% MoS₂, the corrosion inhibition efficiency of the steel sheet was as high as 96.97%. The main reason was that the longer stirring and grinding were conducive to the uniform distribution of MoS₂ in the grease, and the protective film formed by MoS₂ and GCr15-bearing steel improved the lubrication performance and corrosion inhibition performance of the friction system.

Keywords: MoS₂; Lithium base grease; addition method; Friction; corrosion

1. Introduction

Grease is a common lubricant composed of base oil and thickener and is widely used in machinery lubrication due to its unique properties. It is often the lubricant of choice for rolling bearings, plain bearings, slider bearings, gears, pivots, couplings, guides, pin bushings, and sliding contacts [1]. Greases have good sealing ability, are leak-resistant, have corrosion resistance, and require little maintenance [2]. Grease occupies an important position in the national economy and plays an important role in maintaining the normal operation of various mechanical equipment, reducing friction and wear during operation, and extending the service life of mechanical equipment [3].

At present, there are five main types of base oils for the production of biodegradable fats and oils: highly unsaturated vegetable oil, low-viscosity polyalphaolefin, polyethylene glycol, dibasic acid ester, and polyol ester. The main advantages of vegetable oil are low toxicity, high biodegradation rate, low cost, and renewable [4].

In general, various types of greases can be used for bearing lubrication. The low-performance calcium and sodium grease is cheap, but the lubrication effect is not as good as the high-performance grease, and the grease change cycle is short. For bearings of working conditions with general speed, low load, and low working temperature, calcium base or compound calcium base grease can be used. Sodium base grease can be used in environments with slightly higher working temperatures and no water and wet circumstances. Calcium and sodium base grease can be used in environments with water and wet. Ball-bearing grease can be used for equipment with general speed and heavy working load, as its mechanical stability and colloidal stability are better than calcium and sodium base greases[5]. Compared with other soap-based greases, lithium-based greases can still exert excellent lubrication performance under extremely harsh operating conditions.

In the field of lubrication engineering, the tribological behavior of grease is largely determined by the performance of additives. The main function of additives in grease is to improve and enhance the performance [6], and the addition of different additives will lead to differences in the performance of the same grease. For low-speed and heavy load conditions, lithium-based grease containing MoS₂ exhibits good tribological properties. MoS₂ is a two-dimensional layered structure formed by an S-Mo-S atomic covalent bond, and the layers are connected by weak van der Waals forces, and the molecular layers are easy to slide so that they show excellent frictional properties [7], and have been widely used in the field of solid lubrication [8]. In addition, compared with other lubricants, a series of lubricants containing MoS₂ displays tribological advantages. Research has found that MoS₂ is prone to react with non-ferrous metals such as iron substrates, generating friction films containing iron sulfide and its oxides, metal oxides, and disulfides [9], thus exhibiting excellent low friction and wear resistance. Therefore, MoS₂ is commonly used as an additive and is widely used in lubricating oils or greases to reduce friction and wear [10].

2. Experimental Part

2.1. Experimental Materials and Characterization

Main materials and instruments: Molybdenum disulfide (MoS₂, AR), lithium dodecyl stearate, Cornoil, nitrogen, petroleum ether (60-90), anhydrous ethanol (AR), deionized water, three-neck flask, magnetic Stirrer, three-roll grinder (S65, China Changzhou Caobao Machinery Co., LTD.), vertical universal friction and wear testing machine (MMW-1B), GCr15-bearing steel ball (diameter 10mm, hardness HRC62-65).

MoS₂ powder, produced by Tianjin Zhiyuan Chemical Reagent Co., LTD. The morphology of the samples was observed by Zeiss SU8010 scanning electron microscope (SEM). The powder samples were ultrasonically dispersed on the silicon wafer with anhydrous ethanol, and then gold was sprayed after drying. Figure 1 is the SEM image of MoS₂ powder. It can be seen from SEM that MoS₂ is a lamellar structure. Because the layered structure of MoS₂ is only affected by weak van der Waals forces between layers, it is prone to sliding. This results in lubricants containing MoS₂ typically exhibiting good friction reduction and wear resistance [11].

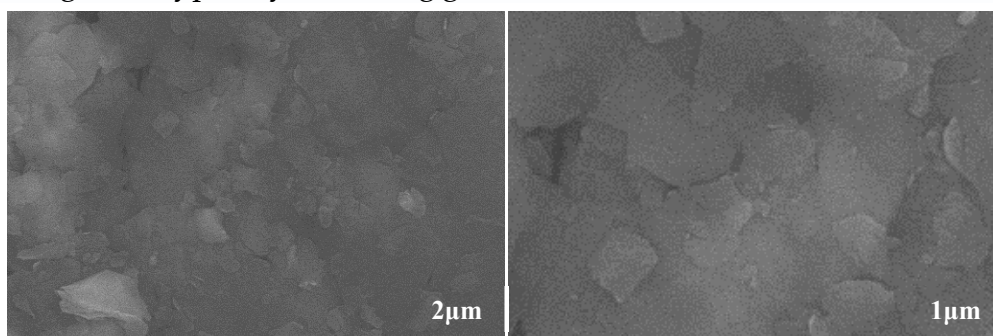


Figure 1. SEM image of MoS₂ powder.

2.2. Preparation of Grease

The specific preparation method of grease is as follows: 150 g of corn oil was heated in an oil bath to 110°C. In the atmosphere of nitrogen, 25 g of thickening agent (12-hydroxystearate lithium) was gradually added to corn oil for thickening treatment. After stirring the reactant for 1 h, the temperature was raised to 180°C for 1h, and finally, the temperature was raised to 210°C for 40 min. After that, the reaction was cooled naturally in the oil bath. The cooled grease was then ground 5-10 times by a three-roll grinder to obtain the samples of the greases used in the following experiments.

In this study, MoS₂ additives with different mass fractions (ω) were added to the prepared grease samples in different ways. The addition methods were divided into the following four types: (1) Adding additives directly during the thickening process, and the method of adding additives was called "Method A". (2) Additives were added during three-roll grinding, this method was referred to as "Method B". (3) After adding additives during the thickening process, the sample was ground with three-rollers for 5-10 times, and the method of adding additives was labeled as "Method C". (4) The MoS₂ additive was added 20 min before thickening, and the resulting grease was ground 5-10 times with a three-roll grinder. This method was recorded as "Method D".

2.3. Performance Test

2.3.1. Physical and Chemical Properties of Grease

Following the test method specified in the GB/T4929 standard, the drop point tester (Shanghai Fine Analysis Instrument Manufacturing Co., LTD., SYD-4929) was used to determine the drop point of the prepared lithium greases. According to the national standard GB/T269, the cone penetration tester (Beijing Luchen Weiye Instrument Equipment Co., LTD., ZRD-3) was used to measure the cone penetration of the prepared lithium greases.

2.3.2. Tribological Properties

The tribological properties of MoS₂ in grease were investigated by a vertical universal friction and wear testing machine. According to the national standard GB-T3142-1982, the high load and high speed test conditions of 392 N, 1450 rpm, and 60 min were adopted, and the friction coefficient was measured and recorded by the mechanical sensor in the friction and wear testing machine in real-time. Before the test, the steel ball and the oil box were ultrasonically cleaned with petroleum ether for 30 min, and the test room temperature was 25±5°C. The experiment was repeated three times under the same conditions to ensure the accuracy of the test results. After the friction experiment, the diameter of the abrasion spot was measured by an optical microscope.

2.4. Analysis of Wore Surfaces

The surface morphology of the steel ball was analyzed by scanning electron microscopy (SEM) of Zeiss SU8010. Before the SEM examination, the test balls were ultrasonically cleaned twice with petroleum ether for about 15 min each time. XPS is a highly sensitive ultramicroscopic surface analysis technology, which can be used to achieve qualitative and semi-quantitative analysis of the composition elements, chemical states, chemical bonds, and charge distribution on the surface of samples according to the peak shape, position, and intensity of different characteristic peaks in XPS spectra. Therefore, XPS was also used for the analysis of worn surfaces in this study. The XPS excitation source used was Al K α rays ($h\nu = 1486.68$ eV), with a working voltage of 15 kV and a filament current of 10 mA. The signal was accumulated for 5-10 cycles. The energy that passed the test was 50 eV, with a step size of 0.05 eV, and the energy of C1s = 284.80 eV was used as the reference standard.

2.5. Corrosion Test

This study investigated the corrosion resistance of lubricating greases prepared by adding different amounts of MoS₂ additives in different ways on iron sheets. The performance of corrosion resistance was quantified by the weight loss of the steel plate.

Before the test, the 20×15×3mm GCr15-bearing steel plate was polished with 320#, 800#, 1000#, 1500# and 2000# sandpaper in turn. Then ultrasonic cleaning was carried out with deionized water and anhydrous alcohol respectively to remove the iron filings on the surface of the steel plate. After the steel plates were naturally air-dried, they were buried in different greases. After 15 days, the steel plates were removed and cleaned with petroleum ether and anhydrous ethanol and then dried in air.

The front and back mass of the steel plate was recorded, and the corrosion resistance of different greases can be analyzed according to the quality difference of each steel plate.

3. Results and Discussion

3.1. Physical and Chemical Properties of Grease

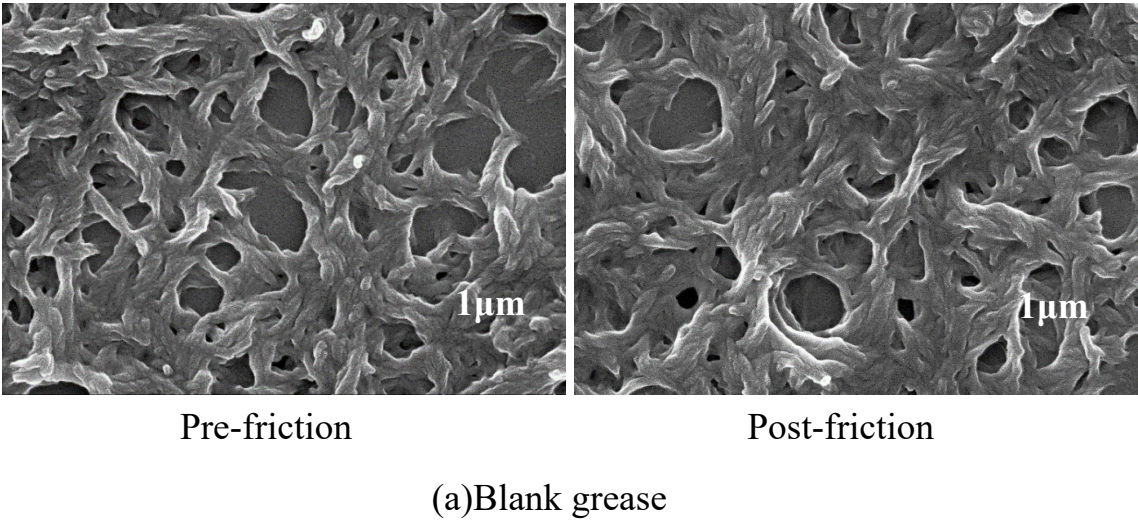
Table 1 shows the drop point and cone penetration of lithium grease after adding MoS₂ additive with different mass fractions in different ways. It can be seen from the data in Table 1 that after adding MoS₂ additives with different mass fractions (ω) in different ways, the drop point of the grease was improved. The coning degree of the prepared lithium grease meets the coning degree requirements of No.4 greases (175~205). Table 1 shows that when MoS₂ was added, the drop point and cone penetration of the grease changed, indicating that the addition of MoS₂ may have a certain effect on the colloidal structure of lithium grease [12].

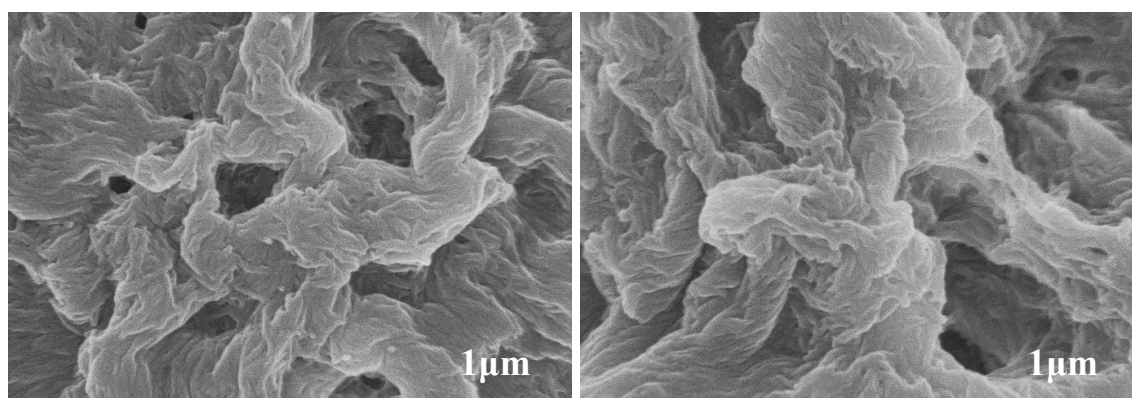
Table 1. Dropping point and cone penetration of greases produced by different addition methods of MoS₂ with different mass fractions (ω).

Addition method		ω (wt%)				
		0	0.01	0.03	0.05	0.07
Dropping point (°C)	Method A	196.5	197	197	202	202
	Method B		202	202	200	199
	Method C		197	197.5	198	198
	Method D		203	199	200	201
Cone penetration (0.1 mm)	Method A	180.6	186.1	202.8	198.1	181.6
	Method B		202.4	201.2	180	192.2
	Method C		175.7	202.3	186.9	180.4
	Method D		184.6	187.5	198.2	180.5

3.2. Soap Fiber Structure of Lithium Lipids

Figure 2 shows the SEM images of the soap fiber structure of Blank grease and lithium-based lubricating grease prepared by adding MoS₂ through the “Method D” under the friction test conditions of 392 N, 1450 r/min, and 60 min before and after friction. By comparison, it can be found that the network structure of soap fiber of blank lithium-based grease was sparse before and after the friction. The more compact the soap fiber structure, the higher the drop point of the grease, the better the colloidal stability[13]. The soap base structure of “Method D” had small pores and strong oil absorption capacity, so it can act on the friction pair for a long time and play a role in reducing friction and anti-wear.





Pre-friction

Post-friction

(b) 0.01wt% MoS₂ by “Method D”

Figure 2. SEM image of soap fiber structure of Blank grease and lithium grease with 0.01wt% MoS₂ by the “Method D” before and after friction. (392 N, 1450 rpm, 60 min).

3.3. Tribological properties

3.3.1. Friction Reduction Performance

Under the experimental conditions of 392 N, 1450 rpm, room temperature, and 60 min, the friction reduction and anti-wear properties of MoS₂ greases prepared in different ways were estimated. Figures 3 and 4 show the friction coefficient relationship curve and average friction coefficient of MoS₂ greases with different mass fractions prepared by different addition methods. As can be seen from the friction coefficient curve of Figures 3(a) and 4(a), when the content of MoS₂ was 0.01% and 0.03%, the friction coefficient relationship curve changes like a peak after a stable period, which may be because the additive content was small, and can not play the role of lubrication for a long time during the friction process. The friction coefficient curve corresponding to 0.05wt% MoS₂ content begins to stabilize after about 500 s, and the average friction coefficient (Figure 3a) was the smallest, with an average friction coefficient of 0.037, indicating that 0.05wt% was the best content when MoS₂ was added by “Method A”.

Figures 3(b) and 4(b) show that when MoS₂ was added by the “Method B”, the average friction coefficient of the prepared grease under experimental conditions first decreased and then increased. By comparing the friction coefficient curves in Figure 3(b), it can be seen that the optimal addition amount of MoS₂ additive in the grease prepared by this method was 0.03wt%. The average friction coefficient of MoS₂ grease at the optimal concentration is 0.040, which decreases by 13.0% compared with the blank group.

The above two methods can improve the anti-friction performance of grease, but “Method B” requires less optimal additive content. The average friction coefficient corresponding to the optimal content of “Method A” is smaller than that of “Method B”. Therefore, “Method C” combines the above two methods.

It can be seen from Figures 3(c) and 4(c) that the friction coefficient of MoS₂ grease prepared by the “Method C” was generally stable under experimental conditions, and gradually decreased over time. The three-roll grinding process in the “Method C” may help the MoS₂ to be more evenly distributed in the grease, thereby reducing friction for longer. By comparing the friction coefficient curves of Figure 3(c), it can be found that the optimal MoS₂ additive content of MoS₂ grease prepared by the “Method C” was 0.05wt%, and the corresponding average friction coefficient was 0.039. When compared with the average friction coefficient of 0.045 in the blank group, it can be found that the friction coefficient was reduced by 15.2%.

“Method C” and “Method D” are different in the addition time of additives. It can be seen from Figures 3(d) and 4(d) that when MoS₂ was added by the “Method D”, the friction coefficient curves of MoS₂ grease with different mass fractions were stable, and the optimal content of MoS₂ corresponding to this method was 0.01wt%, and its average friction coefficient was 0.034. This was a 26.1% reduction compared to the blank group.

Compared with four different MoS₂ adding methods, it can be found that the friction coefficient of grease obtained by the “Method D” was the most stable, the average friction coefficient was reduced the most, and the required optimal MoS₂ content was lowest. This may be due to the longest reaction time between the additive and the base oil, which was more conducive to the uniform mixing of the additive in the grease so that the friction reduction effect can be effectively achieved for a longer time. The lubricating grease prepared by this method can achieve a good lubrication effect based on a very low additive content of 0.01wt%.

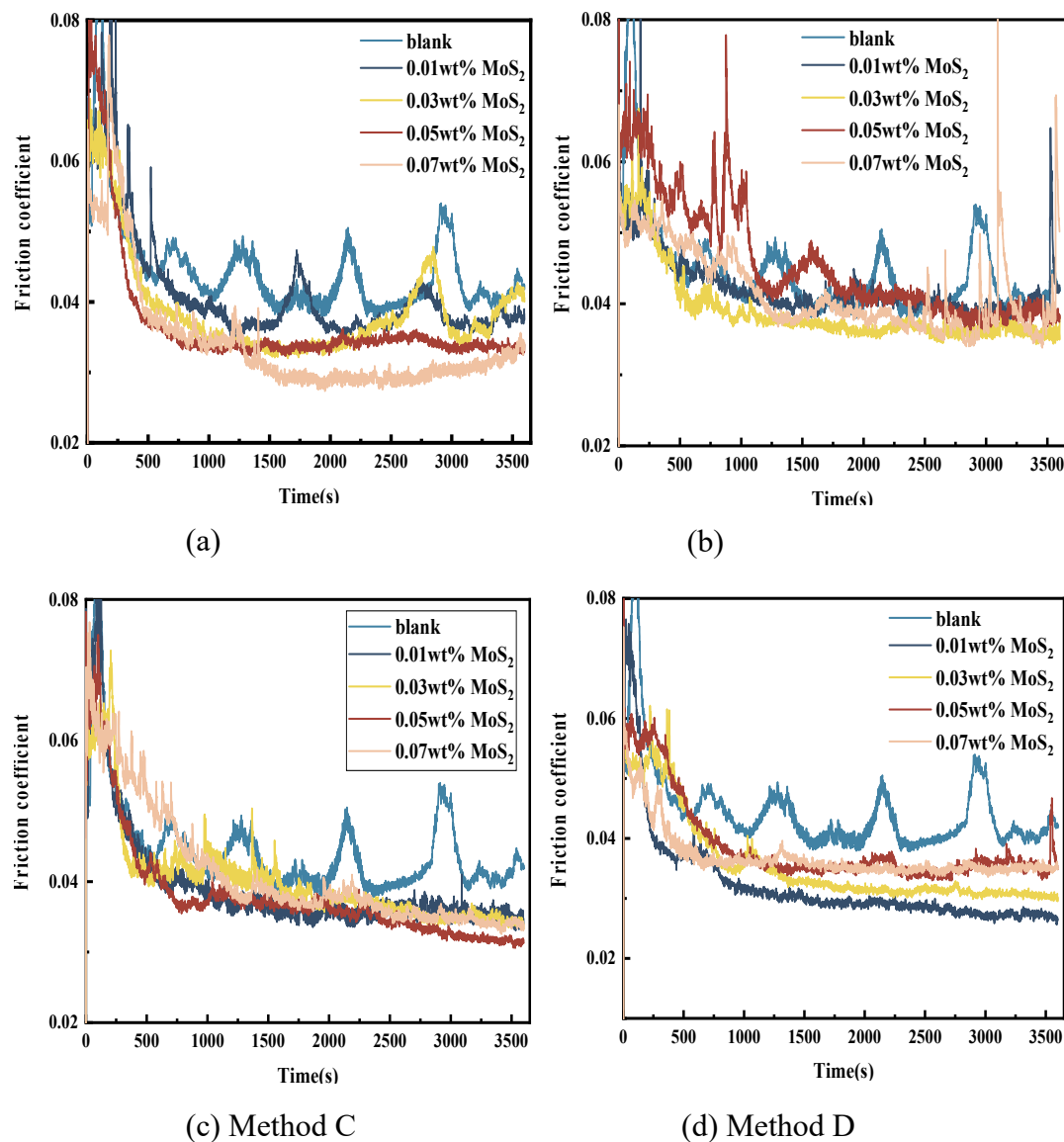


Figure 3. Friction coefficient curves of MoS₂ greases prepared by different addition methods with variable amounts of MoS₂. (a) Method A, (c) Method C, (b) Method B, (d) Method D. (392 N, 1450 rpm, 60 min).

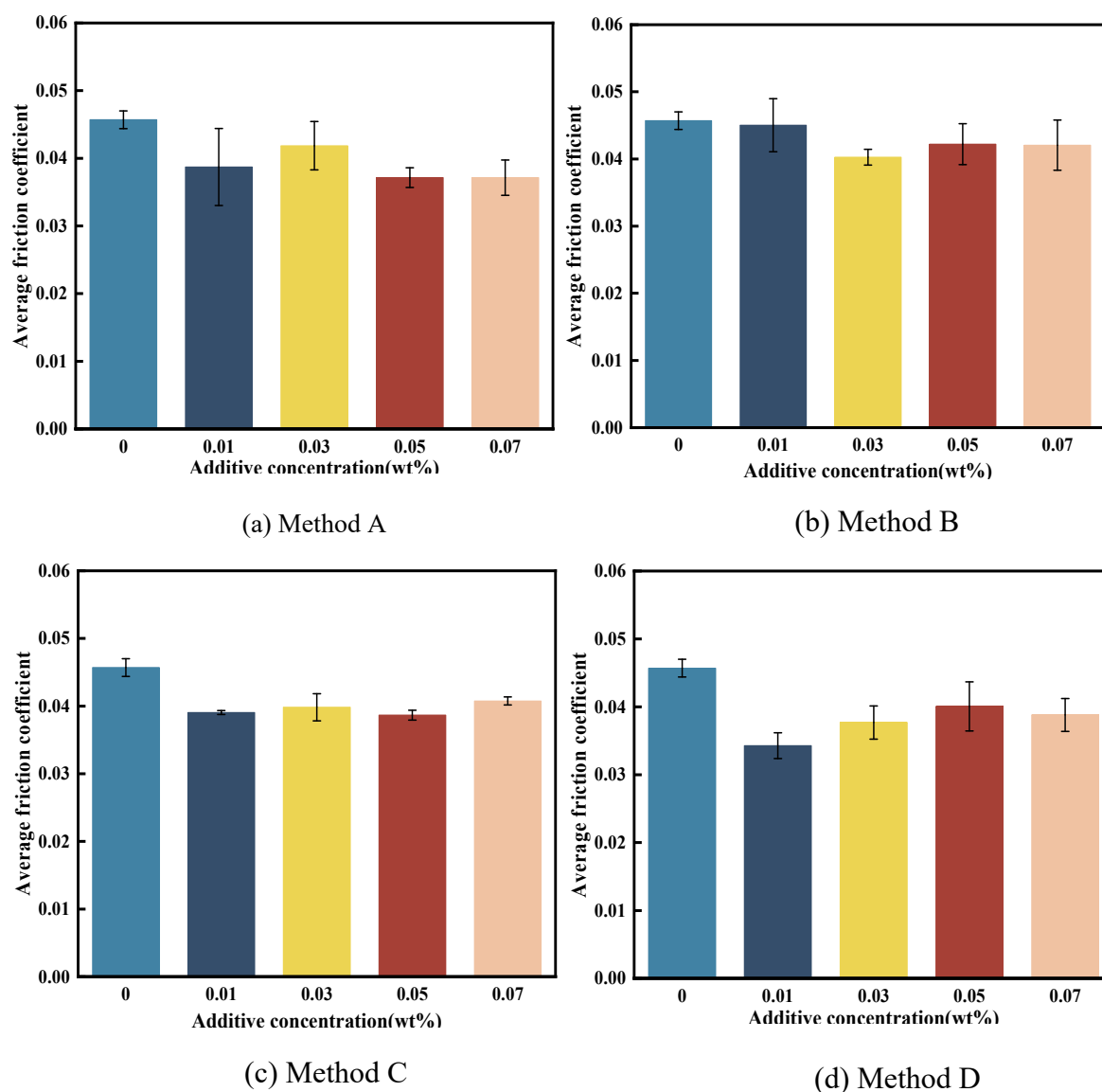


Figure 4. Friction coefficient of MoS₂ greases prepared by different addition methods with variable amounts of MoS₂. (a) Method A, (b) Method B, (c) Method C, (d) Method D. (392 N, 1450 rpm, 60 min).

3.3.2. Anti-Wear Performance

Figure 5 shows the average wear diameter of MoS₂ greases with different mass fractions prepared by different addition methods under the test conditions of 392 N and 1450 rpm. From Figure 5a, it can be seen that the average wear spot diameter corresponding to the lubricating grease prepared by the “Method A” shows that the optimal MoS₂ content was 0.05wt%, at which point the minimum average wear spot diameter was 0.50 mm. From Figure 5b, it can be seen that the wear spot diameter caused by MoS₂ lubricating grease prepared by the “Method B” shows a trend of first decreasing and then increasing with the increase of MoS₂ content, and the optimal content of additives required for the lubricating grease prepared by this method was 0.03wt%. From the average wear spot diameter in Figure 5c, it can be observed that the optimal amount of MoS₂ added to the lubricating grease prepared by the “Method C” was also 0.05wt%. When the MoS₂ content was 0.01wt%, a small amount of MoS₂ could not effectively resist wear during the friction process. When the amount of MoS₂ exceeded 0.03wt%, due to the low surface tension and hydrophilic properties of the MoS₂ sliding layer [14], MoS₂ particles may be unevenly dispersed in the lubricating grease, resulting in a gradual increase in wear spot diameter. Figure 5d shows the corresponding wear spot

diameter of the lubricating grease prepared using the “Method D”. From Figure 5d, it can be seen that the lubricating grease configured with 0.01wt% MoS₂ exhibits the smallest average wear spot diameter, which was reduced by 0.16 mm compared to the blank group without MoS₂.

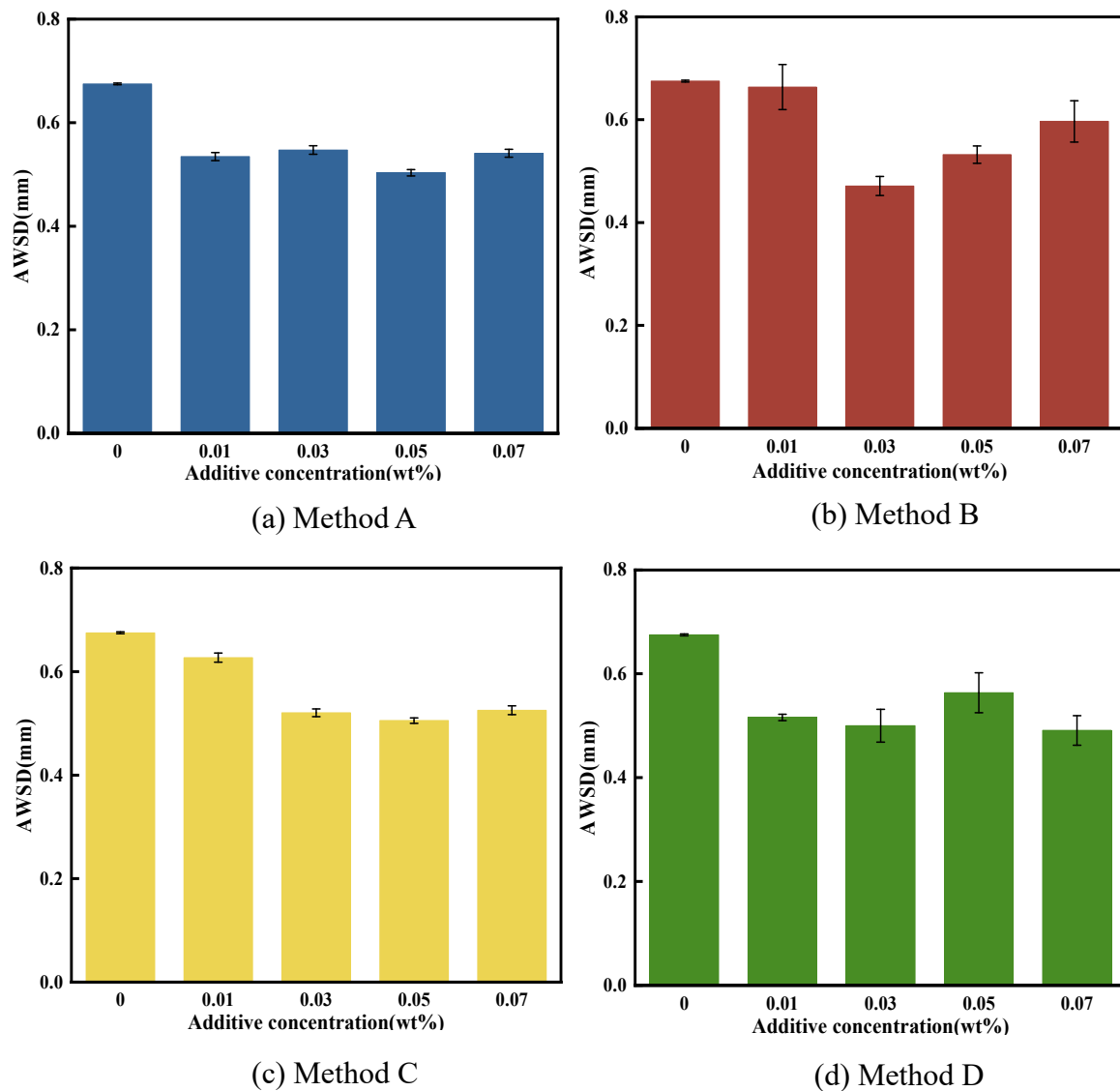


Figure 5. Wear spot diameter of MoS₂ greases prepared by different addition methods with variable amounts of MoS₂. (a) Method A, (b) Method B, (c) Method C, (d) Method D. (392 N, 1450 rpm, 60 min).

3.4. SEM Analysis of Worn Surfaces

Figure 6 shows the SEM images of wear spots corresponding to blank lubricating grease and lubricating grease with the optimal mass fraction of MoS₂ added in different ways. From Figure 6a, it can be seen that the wear surface corresponding to the blank grease exhibits obvious friction marks, with wide and large furrows on the wear marks. The wear marks on the steel ball surface (Figures 6b-e) corresponding to the lubricating grease containing the MoS₂ additive become relatively less obvious, and the surface becomes smoother. This may be because MoS₂ has a layered structure that is easy to slide, so it can form a stable film on the rubbing surface under friction conditions, thereby achieving an anti-wear effect.

By comparing Figures 6(b-e), it can be found that the MoS₂ grease prepared by the “Method D” shows the shallowest wear marks and the smoothest grinding surface, indicating that the grease prepared by this addition method has the best anti-wear effect. This may be because the addition of

MoS₂ before thickening allows MoS₂ and the base oil to be in contact and mixed for a longer period, and may also have a longer chemical reaction. Moreover, this method adopted a three-roller grinder for grinding, which allows MoS₂ to be more evenly distributed in the lubricating grease. Therefore, the MoS₂ lubricating grease prepared by the “Method D” exhibits the smallest wear spot diameter and the least obvious friction marks.

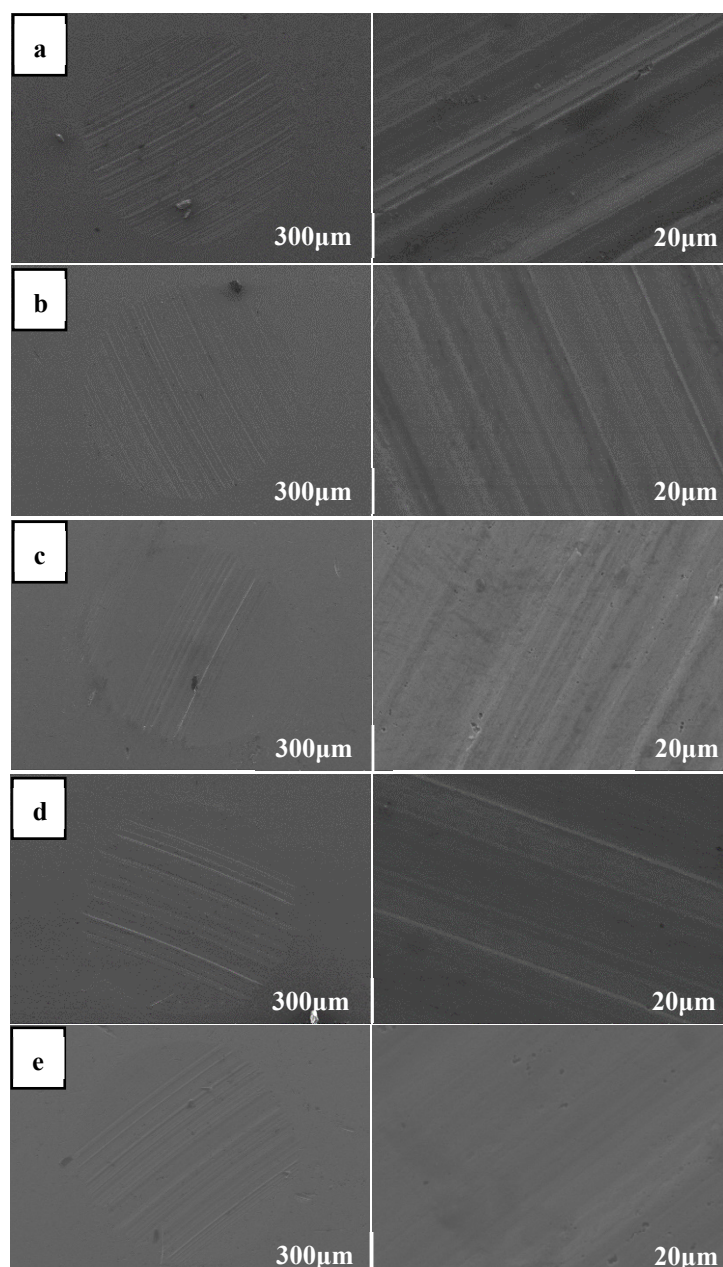


Figure 6. SEM images of worn surfaces (a) blank group, (b) Method A with 0.05wt% MoS₂, (c) Method B with 0.03wt% MoS₂, (d) Method C with 0.05wt% MoS₂, (e) Method D with 0.01wt% MoS₂. (392 N, 1450 rpm, 60 min).

3.5. XPS Analysis of Worn Surfaces

In order to reveal the lubrication mechanism of MoS₂ as an additive in lithium grease, the chemical state of elements on the surface of the abrasive spot was analyzed by XPS. Figure 7 shows the XPS spectra of C1s, O1s, Fe2p, S2p, and Mo3d on the wear surface of steel balls caused by lithium-based grease with 0.01% MoS₂.

The peaks with binding energies of 284.8, 284.98 and 287.18 eV in the C1s spectra (Figure 7b) correspond to C-C, C-O-C [15] and C=O [16] bonds, respectively. The binding energies of O1s (Figure

7c) at 529.37 and 531.81 eV peaks correspond to metal oxides (Fe_2O_3) and C-O bonds, respectively [17]. The binding energies of Fe2P (Figure 7d) at 706.30 and 711.76 eV peaks correspond to FeS_2 and Fe_2O_3 , respectively[18]. The 709.64 and 723.19 eV correspond to Fe $2\text{P}_{3/2}$ and Fe $2\text{P}_{1/2}$ of FeO, respectively. The peaks of the S2p-XPS spectra (Figure 7e) at 161.50 and 168.60 eV correspond to MoS_2 [19]/ FeS_2 and Metal sulfate, respectively. The peaks at 232.68 and 235.83 eV in the Mo3d-XPS spectra (Figure 7f) correspond to MoO_2 and MoO_3 , respectively[20]. The appearance of MoO_2 and MoO_3 indicates that the MoS_2 additive has been oxidized during the friction process, that is, MoS_2 may partially undergo tribochemical reactions during the friction process.

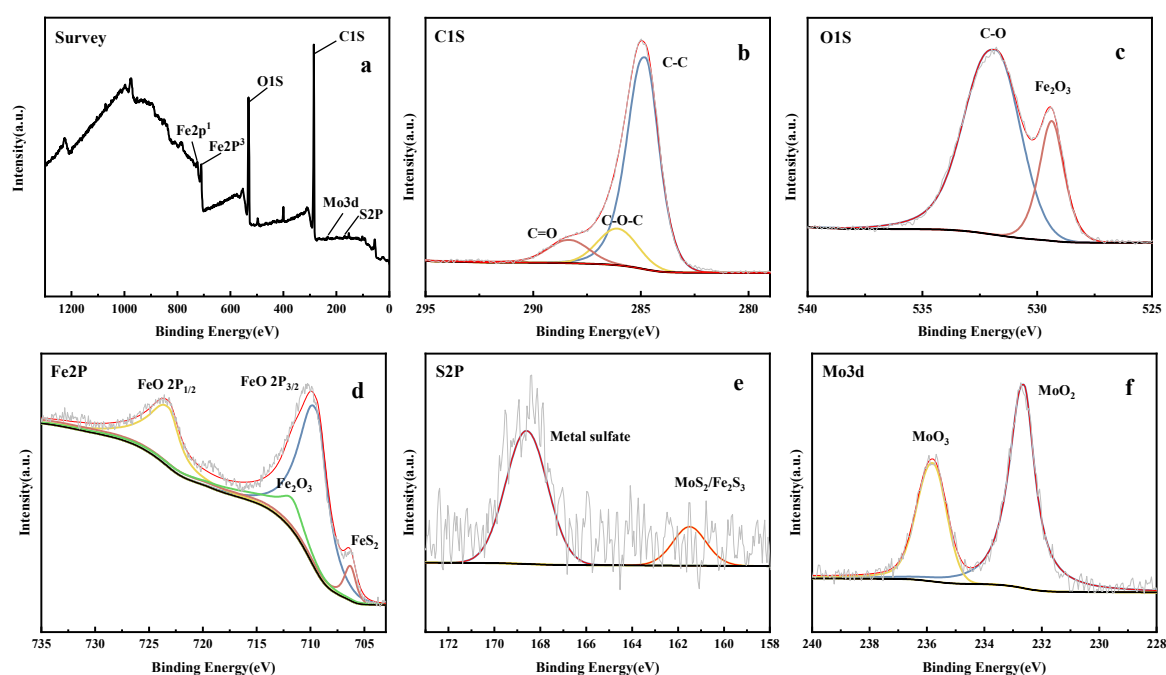


Figure 7. XPS spectra of the worn surface caused by 0.01wt% MoS_2 lithium grease prepared by "Method D".

3.6. Results of Corrosion Test

To explore the corrosion performance of MoS_2 -lithium grease on metal under natural conditions, the corrosion test was conducted with the GCr15-bearing steel sheet. Table 2 shows the quality changes of the steel sheets before and after 15 days.

Table 2. Mass change (Δm , g) of GCr15-bearing steel plate after 15 days of exposure to MoS_2 lubricating grease.

Addition method	Mass change (Δm , g)				
	ω (wt%)				
	0	0.01	0.03	0.05	0.07
Method A	0.0198	0.0034	0.0109	0.0059	0.0040
Method B	0.0198	0.0006	0.0083	0.0034	0.0070
Method C	0.0198	0.0089	0.0032	0.0089	0.0017
Method D	0.0198	0.0161	0.0158	0.0167	0.0177

The corrosion rate of MoS_2 grease on bearing steel can be calculated according to the following formula [21]:

$$CR = \frac{\Delta m}{At} \quad (3-1)$$

$$\eta_{wL} = \frac{CR_0 - CR_1}{CR_0} \times 100\% \quad (3-2)$$

Where Δm represents weight loss, A is the surface area of the bearing steel, t is the time of the steel plate buried in the grease, CR represents the corrosion rate, CR_0 and CR_1 represent the corrosion rate with and without additives, respectively.

Figure 8 shows the corrosion rate of GCr15-bearing steel sheets buried in MoS₂ lubricating grease with different mass fractions prepared by different addition methods for 15 days. Compared with the blank group without MoS₂, it can be found that the addition of MoS₂ additive reduces the corrosion rate of bearing steel in the natural environment, indicating that the addition of MoS₂ effectively slows down the corrosion process of bearing steel.

However, as the addition of additives changes, the content of MoS₂ with the best corrosion inhibition efficiency also changes. The optimal corrosion inhibition efficiency of MoS₂ by the "Method A" was found at 0.01wt% MoS₂ content. When MoS₂ was added by the "Method B", the optimal corrosion inhibition efficiency occurred when the MoS₂ content was 0.01wt%. When MoS₂ was added by the "Method C", the optimal additive content for corrosion inhibition efficiency was 0.07wt%. When the "Method D" was adopted to add MoS₂, 0.03wt% MoS₂ exhibited the best corrosion inhibition efficiency. This change may be due to the varying degree of uniform distribution of MoS₂ in the lubricating grease when added in different ways, resulting in different corrosion results. The data in Table 2 and Figure 8 both show that when the "Method B" was used to add 0.01wt% MoS₂, the configured grease had the lowest corrosion rate on the bearing steel, that is, the corresponding corrosion inhibition efficiency was the highest, reaching 96.97%.

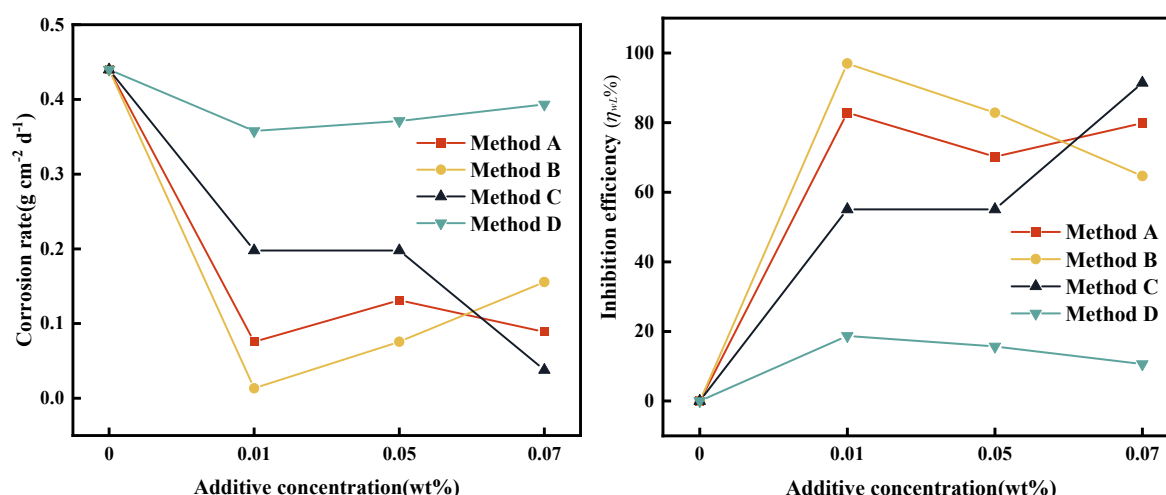


Figure 8. Corrosion rate and corrosion inhibition efficiency of GCr15-bearing steel plate buried in MoS₂ greases with different mass fractions prepared by varying addition methods.

The SEM image of the GCr15-bearing steel sheet embedded in 0.01% MoS₂ lubricating grease for 15 days is shown in Figure 9. Figure 9a shows that no obvious surface coverings or corrosion pits were found on the surface of the original bearing steel sheet. However, the surface of the bearing steel corresponding to the blank grease without MoS₂ showed obvious corrosion, with an unsmooth surface and severe corrosion pits (Figure 9b). Figure 9c is the SEM diagram of the surface of the steel sheet caused by 0.01wt% MoS₂ grease prepared by the "Method A". It can be seen that corrosion pits appear in a small part of the surface of the steel sheet under such circumstances. When 0.01wt% MoS₂ was added using the "Method B", the resulting grease causes the slightest corrosion on the surface of the steel sheet (Figure 9d). When 0.01wt% MoS₂ grease was prepared by "Method C", the corrosion test results in uneven corrosion on the surface of the steel sheet, as shown in Figure 9e. As can be seen from Figure 9f, the grease prepared by adding MoS₂ additive with the method of "Method D" will cause local corrosion on the surface of the steel sheet. From Figures 9 (c-f), it can be found that MoS₂ grease has a certain corrosion effect on the steel surface, which may be because the S element in MoS₂

can combine with the Fe element in the bearing steel to produce iron sulfide and other products [9], so MoS₂ has a certain corrosion effect, and the surface of the steel sheet was therefore pitted. However, by comparing Figure 9b and Figures 9 (c-f), it can be found that after the addition of MoS₂ additive, the corrosion phenomenon on the surface of the bearing steel was slowed down in different forms, indicating that MoS₂ has a certain corrosion inhibition effect.

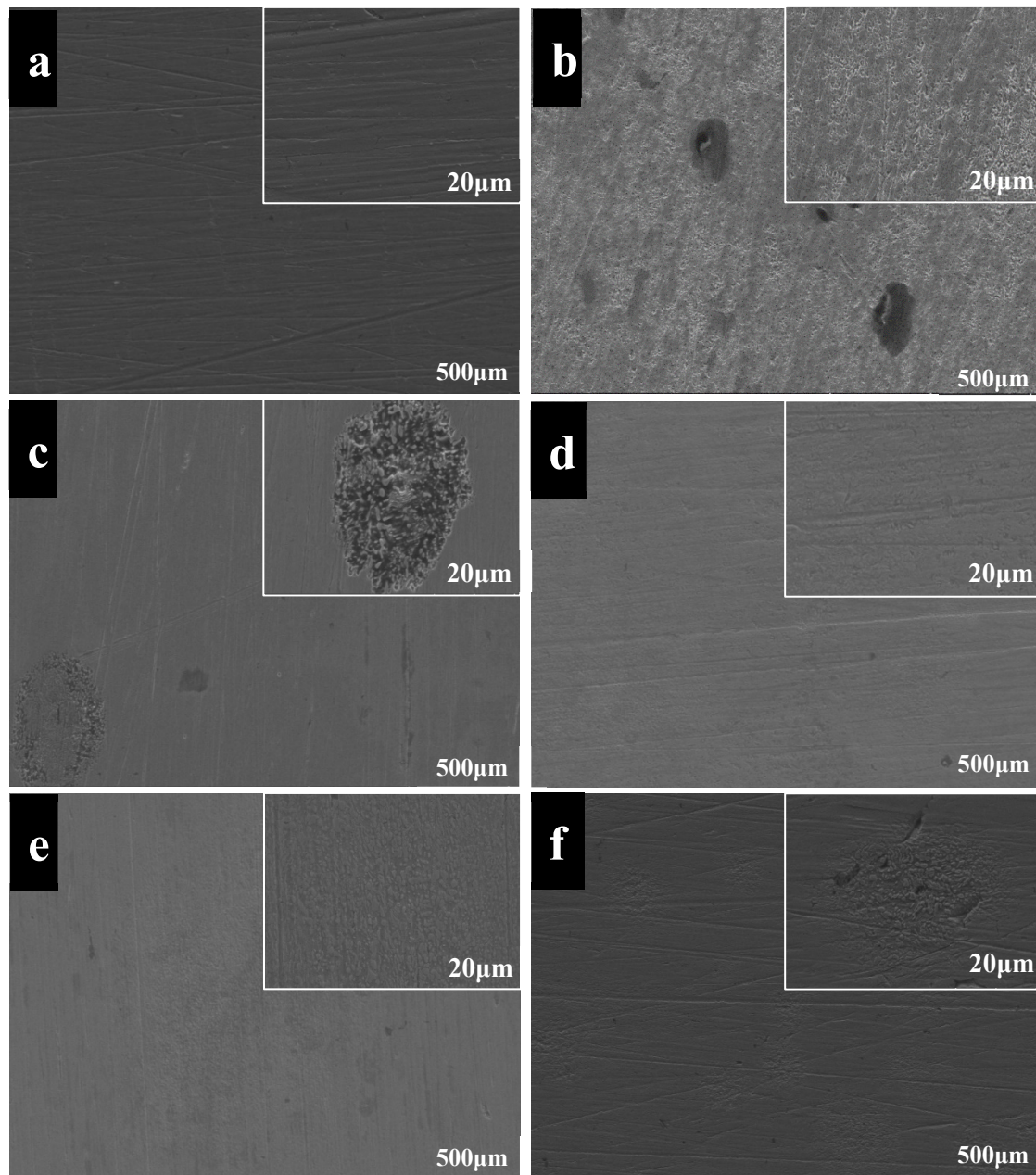


Figure 9. SEM image of GCr15 steel sheet embedded with 0.01wt% MoS₂ lubricating grease prepared by different addition methods after 15 days (a) original steel surface (b) blank grease (c) Method A (d) Method B (e) Method C (f) Method D.

To better demonstrate the morphology and roughness of GCr15-bearing steel sheets after corrosion experiments, three-dimensional (3D) morphology studies were also conducted on the corroded steel sheets. Figure 10 shows the 3D morphology of the steel sheet after being embedded in 0.01% MoS₂ grease for 15 days.

From Figure 10a, it can be observed that the surface roughness of the original bearing steel was 0.034 µm. After 15 days of embedding with blank lubricating grease without MoS₂, the surface

roughness of the bearing steel was the highest, reaching $0.223\text{ }\mu\text{m}$ (Figure 10b). Figure 10c illustrates that the surface roughness of the corroded steel plate corresponding to the lubricating grease prepared by the "Method A" was $0.113\text{ }\mu\text{m}$. Figure 10d shows that the roughness of the corroded steel surface corresponding to the lubricating grease prepared by the "Method B" was $0.047\text{ }\mu\text{m}$. Figure 10e and 10f represent the 3D images of the corroded steel surface corresponding to the lubricating grease prepared by the "Method C" and the "Method D", respectively, with a surface roughness of $0.193\text{ }\mu\text{m}$ and $0.054\text{ }\mu\text{m}$.

Comparing Figure 10b and Figures 10 (c-f), it can be found that regardless of the method of adding $0.01\text{wt}\%$ MoS_2 , the formulated lubricating grease will reduce the surface roughness of bearing steel after corrosion. The adoption of the "Method B" resulted in the maximum reduction of surface roughness (Figure 10d) by $0.176\text{ }\mu\text{m}$. The method of "Method C" (Figure 10e) with the least reduction in surface roughness resulted in a reduction of only $0.03\text{ }\mu\text{m}$. The 3D morphology in Figure 10 is in good agreement with the SEM results in Figure 9. This further indicates that adding MoS_2 additive can slow down the corrosion rate of bearing steel and can protect the corrosion of the device for a long time under natural conditions.

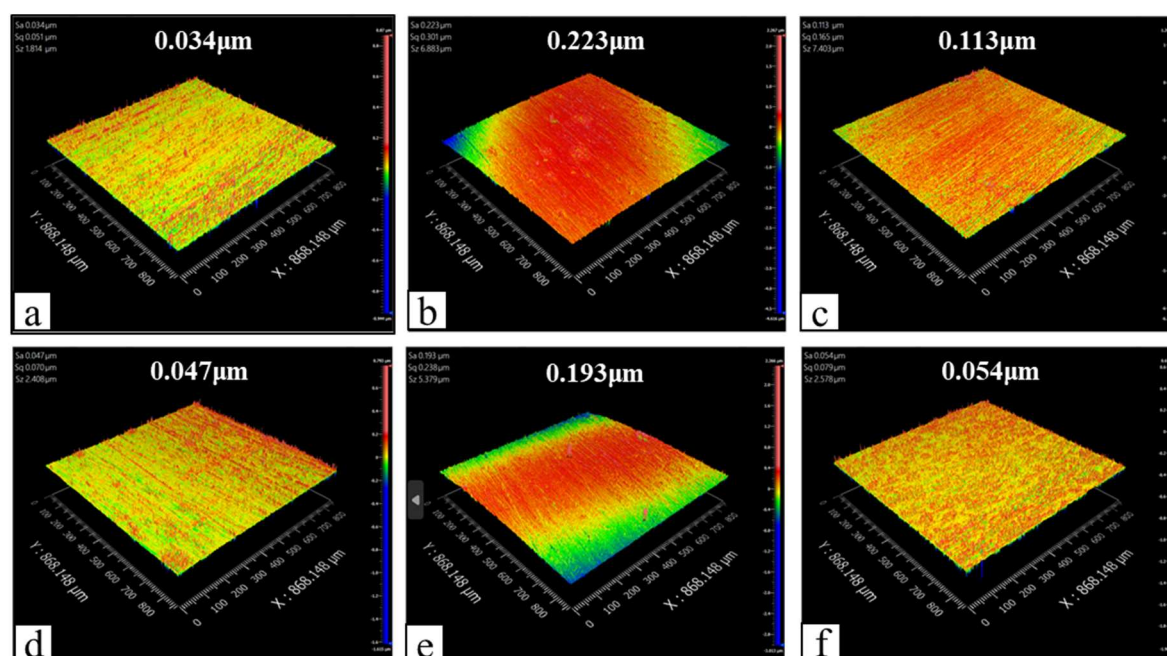


Figure 10. 3D morphology of GCr15-bearing steel sheet embedded in $0.01\text{wt}\%$ MoS_2 lubricating grease prepared by different methods for 15 days (a) original steel sheet, (b) blank lubricating grease without MoS_2 , (c) Method A, (d) Method B, (e) Method C, (f) Method D.

4. Conclusions

The effects of MoS_2 addition methods on the tribological and corrosion properties of MoS_2 -containing lithium grease were investigated in this study. Based on the above analysis, the following conclusions can be drawn:

1. When MoS_2 was used as an additive in lithium-based greases, it exhibited ideal friction reduction and anti-wear effects and corrosion inhibition performance.
2. The content of additive MoS_2 was 0.01% , the friction coefficient of "Method D" was the most stable, the average friction coefficient was 0.034 , and the average spot diameter was reduced by 0.16mm . "Method B" had the highest corrosion inhibition efficiency (96.97%).
3. The soap base porosity of "Method D" is small, and the ability to wrap the base oil and MoS_2 is strong, so that it can play the role of anti-friction and anti-wear for a long time. MoS_2 lithium grease has good anti-corrosion properties, which may be "Method B" can evenly distribute MoS_2 on the

surface of the grease, so that the protective film formed by MoS₂ and GCr15-bearing steel can effectively slow down the corrosion effect of lithium-based grease on steel.

Acknowledgments: The authors acknowledge the financial support provided by the National Natural Science Foundation of China (51965020; 52201160); Jiangxi Natural Science Foundation of China (20232ACB204001; 20232BAB204008; 20232BAB212007; 20212BBE53041, 20224BAB204048, 20224ACB204014).

References

1. Lugt, P. A Review on Grease Lubrication in Rolling Bearings. *Tribology Transactions* **2009**, *52*, 470-480, doi:https://doi.org/10.1080/10402000802687940.
2. Gurt, A.; Khonsari, M. The Use of Entropy in Modeling the Mechanical Degradation of Grease. *Lubricants* **2019**, *7*, doi:https://doi.org/10.3390/lubricants7100082.
3. Jiang, M.; Guo, X.; Dong, J.; Chen, G. Study on the lithium complex grease. *Lubrication Engineering(China)* **2000**, 25-28.
4. Lligadas, G.; Ronda, J.C.; Galià, M.; Cádiz, V. Renewable polymeric materials from vegetable oils: a perspective. *Materials Today* **2013**, *16*, 337-343, doi:https://doi.org/10.1016/j.mattod.2013.08.016.
5. Chen, X.; Chen, G. Analysis of lithium molybdenum disulfide base grease and its application in roll bearing. *Science & Technology Information(China)* **2013**.
6. Cheng, S.; Guo, X.; Jiang, M.; He, Y. Research progress of lithium complex grease. *Contemporary Chemical Industry(China)* **2018**, *47*, 152-158, doi:https://doi.org/10.13840/j.cnki.cn21-1457/tq.2018.01.039.
7. Rapoport, L.; Bilik, Y.; Feldman, Y.; Homyonfer, M.; Cohen, S.R.; Tenne, R. Hollow nanoparticles of WS₂ as potential solid-state lubricants. *Nature* **1997**, *387*, doi:https://doi.org/10.1038/42910.
8. Li, J.; Wang, Y.; Liu, D.; He, K.; Lu, L. Research progress on the properties and application of molybdenum disulfide. *Powder Metallurgy Technology(China)* **2021**, *39*, 471-478, doi:10.19591/j.cnki.cn11-1974/tf.2021020008; http://pmt.ustb.edu.cn.
9. Niste, V.B.; Ratoi, M. Tungsten dichalcogenide lubricant nanoadditives for demanding applications. *Materials Today Communications* **2016**, *8*, 1-11, doi:https://doi.org/10.1016/j.mtcomm.2016.04.015.
10. Wu, Z. Synthesis of molybdenum (tungsten) disulfide nanostructures and their properties[D]. Central South University, **2012**.
11. Cheng, Y. Preparation and tribological properties of space lubricating grease containing MoS₂ nanoparticles[D]. Hefei University of Technology, **2012**.
12. Wang, W.; Tian, S.; Sun, H. Influence of base oil and additives on microstructure of grease. *Petroleum Products Application Research(China)* **2015**, *33*, 26-34, doi:https://doi.org/10.3969/j.issn.1006-1479.2015.05.003.
13. Xie, X. Experimental study on soap fiber structure affection the performance of grease[D]. Harbin Institute of Technology, **2008**.
14. Yuan, s. Study on the surface modification and friction properties of molybdenum disulfide. *Modern Salt and Chemical Industry(China)* **2019**, *46*, doi:https://doi.org/10.19465/j.cnki.2095-9710.2019.01.022.
15. Kosynkin, D.V.; Higginbotham, A.L.; Sinitskii, A.; Lomeda, J.R.; Dimiev, A.; Price, B.K.; Tour, J.M. Longitudinal unzipping of carbon nanotubes to form graphene nanoribbons. *Nature* **2009**, *458*, 872-876, doi:https://doi.org/10.1038/nature07872.
16. Liang, Y.; He, X.; Chen, L.; Zhang, Y. Preparation and characterization of TiO₂-Graphene@Fe₃O₄ magnetic composite and its application in the removal of trace amounts of microcystin-LR. *RSC Advances* **2014**, *4*, 56883-56891, doi:https://doi.org/10.1039/C4RA08258C.
17. Horikawa, T.; Sakao, N.; Sekida, T.; Hayashi, J.i.; Do, D.D.; Katoh, M. Preparation of nitrogen-doped porous carbon by ammonia gas treatment and the effects of N-doping on water adsorption. *Carbon* **2012**, *50*, 1833-1842, doi:https://doi.org/10.1016/j.carbon.2011.12.033.
18. Xiang, S.; Long, X.; Zhang, Q.; Ma, P.; Yang, X.; Xu, H.; Lu, P.; Su, P.; Yang, W.; He, Y. Enhancing Lubrication Performance of Calcium Sulfonate Complex Grease Dispersed with Two-Dimensional MoS₂ Nanosheets. *Lubricants* **2023**, *11*, 336, doi:https://doi.org/10.3390/lubricants11080336.
19. Fan, X.; Li, W.; Li, H.; Zhu, M.; Xia, Y.; Wang, J. Probing the effect of thickener on tribological properties of lubricating greases. *Tribol. Int.* **2018**, *118*, 128-139, doi:https://doi.org/10.1016/j.triboint.2017.09.025.

20. Jiang, H.; Hou, X.; Ma, Y.; Su, D.; Qian, Y.; Ahmed Ali, M.K.; Dearn, K.D.J.W. The tribological performance evaluation of steel-steel contact surface lubricated by polyalphaolefins containing surfactant-modified hybrid MoS₂/h-BN nano-additives. *Wear* **2022**, 504–505, doi:<https://doi.org/10.1016/j.wear.2022.204426>.
21. Zhu, Y.; Sun, Q.; Wang, Y.; Tang, J.; Wang, Y.; Wang, H. Molecular dynamic simulation and experimental investigation on the synergistic mechanism and synergistic effect of oleic acid imidazoline and l-cysteine corrosion inhibitors. *Corrosion Science* **2021**, 185, 109414, doi:<https://doi.org/10.1016/j.corsci.2021.109414>.

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.