lubricants

Article

Study on the Influence of the MoS_2 Addition Method on the Tribological and Corrosion Properties of Greases

Can Zhu [1], Zhongyi He [1,*], Liping Xiong [1], Jiusheng Li [2], Yinglei Wu [3,*] and Lili Li [1]

[1] School of Materials Science and Engineering, East China Jiaotong University, Nanchang 330013, China; zczy990117@163.com (C.Z.); helijia666@163.com (L.X.); lilshd18@gmail.com (L.L.)
[2] Laboratory of Advanced Lubricating Materials, Shanghai Advanced Research Institute, Chinese Academy of Sciences, Shanghai 201210, China; lijs@sari.ac.cn
[3] School of Chemistry and Chemical Engineering, Shanghai University of Engineering Science, Shanghai 201210, China
* Correspondence: hzy220567@163.com (Z.H.); wuyl@sues.edu.cn (Y.W.)

Abstract: MoS_2 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 MoS_2 additive on its tribology and corrosion properties. In this work, eco-friendly vegetable oil was selected as the base oil, with MoS_2 powder as the additive to synthesize lithium-based grease. The effects of different adding modes of MoS_2 on the tribology and corrosion properties of the grease were studied. The experimental results showed that adding 0.01 wt% MoS_2 before thickening (Method D) was more conducive to improving the tribological properties of lithium grease. The average friction coefficient was reduced by 26.1%, and the average wear scar diameter was reduced by 0.16 mm. After grinding and adding (Method B) 0.01 wt% MoS_2, the corrosion inhibition efficiency of the steel sheet was as high as 96.97%. The reason was that the tribochemical reaction of MoS_2 evenly distributed throughout the grease during friction, forming a thin friction film, reducing friction and wear. The protective film formed by MoS_2 and GCr15-bearing steel improved the corrosion inhibition performance of the grease.

Keywords: MoS_2; lithium-base grease; addition method; friction; corrosion

Citation: Zhu, C.; He, Z.; Xiong, L.; Li, J.; Wu, Y.; Li, L. Study on the Influence of the MoS_2 Addition Method on the Tribological and Corrosion Properties of Greases. *Lubricants* **2023**, *11*, 517. https://doi.org/10.3390/lubricants11120517

Received: 13 November 2023
Revised: 1 December 2023
Accepted: 5 December 2023
Published: 8 December 2023

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 a good sealing ability, are leak-resistant, have corrosion resistance, and require little maintenance [2]. Grease occupies an important position in the global 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 [4], 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 [5].

In general, various types of greases can be used for bearing lubrication [6]. The low-performance calcium and sodium grease is cheap [7], but the lubrication effect is not as good as the high-performance grease, and the grease change cycle is short. Ball-bearing grease can be used for equipment with general speed and heavy working load. Its mechanical stability and colloidal stability are better than calcium and sodium base greases [8]. Compared with other soap-based greases, lithium-based greases can still exert excellent lubrication performance under extremely harsh operating conditions. Lithium-based greases improve the reliability and durability of mechanical components by improving lubrication performance, reducing friction, reducing wear on mating surfaces, and preventing sintering [9–11].

MoS_2 is a two-dimensional layered structure formed by an S-Mo-S atomic covalent bond; the layers are connected by weak van der Waals forces; the molecular layers are easy to slide so that they show excellent frictional properties [12] and have been widely used in the field of solid lubrication [13]. In addition, compared with other lubricants, a series of lubricants containing MoS_2 displays tribological advantages. Research has found that MoS_2 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 [14], thus exhibiting excellent low friction and wear resistance. Therefore, MoS_2 is commonly used as an additive and is widely used in lubricating oils or greases to reduce friction and wear [15,16].

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 [17], and the addition of different additives will lead to differences in the performance of the same grease. Additives are generally added during the preparation of the grease [18] or are directly mixed with the additive. However, the effect of additive adding method has not been reported. In this paper, the effects of trace amounts of MoS_2 addition methods on the tribology and corrosion properties of lithium grease were studied by us.

2. Experimental Part

2.1. Experimental Materials and Characterization

Thickening agent (lithium dodecyl stearate, white powder) purchased from Xinwang Chemical Co., Ltd. (Huzhou, China). Longevity Flower corn oil (edible grade) purchased from Samsung Corn Industry Technology Co., Ltd. (Binzhou, China) was employed as a base oil. Petroleum Ether (60–90) and ethyl alcohol (AR) were purchased from Xilong Science Co., Ltd. (Shantou, China).

MoS_2 powder (AR), produced by Tianjin Zhiyuan Chemical Reagent Co., Ltd. (Tianjin, China). The morphology of the samples was observed by Hitachi SU8010 scanning electron microscope (SEM, HitachiHigh-Technologies Corporation, Tokyo, Japan). The powder samples were ultrasonically dispersed on the silicon wafer with anhydrous ethanol, and then gold was sprayed after drying. Figure 1 shows the SEM image of MoS_2 powder. It can be seen from SEM that MoS_2 is a lamellar structure. Because the layered structure of MoS_2 is only affected by weak van der Waals forces between layers, it is prone to sliding. This results in lubricants containing MoS_2 typically exhibiting good friction reduction and wear resistance [19].

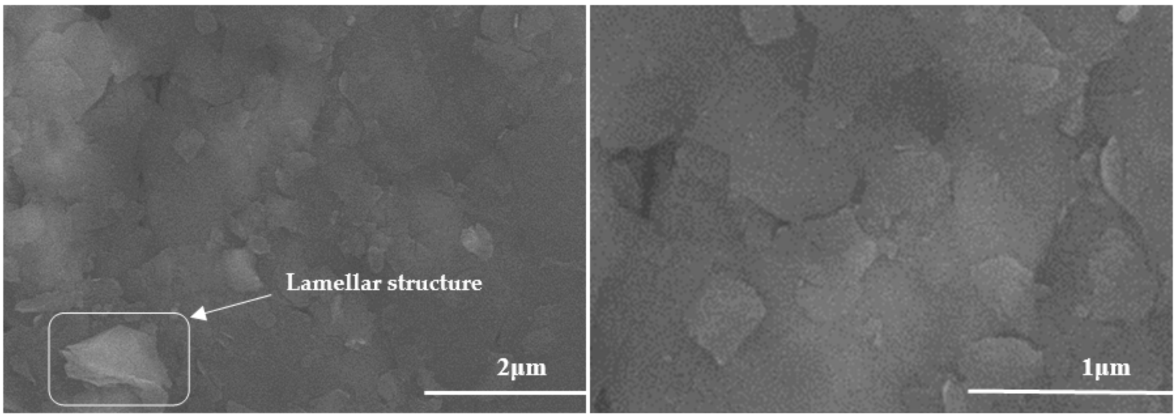


Figure 1. SEM image of MoS_2 powder.

2.2. Preparation of Grease

Based on the preparation method of He et al. [20], 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 1 h, and finally, the reaction temperature was raised to 210 °C until the magnetic stirrer could not stir the grease, then mechanical stirring was used for 1 h. 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 (S65, China Changzhou Caobao Machinery Co., Ltd., Changzhou, China) to obtain the samples of the greases used in the following experiments. The additive concentration of MoS_2 were 0.01 wt%, 0.03 wt%, 0.05 wt% and 0.07 wt%.

In this study, MoS_2 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; 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; the method of adding additives was labeled as "Method C". (4) The MoS_2 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 [21], a drop point tester (Shanghai Fine Analysis Instrument Manufacturing Co., Ltd., SYD-4929, Shanghai, China) was used to determine the drop point of the prepared lithium greases. According to the national standard GB/T269 [22], a cone penetration tester (Beijing Luchen Weiye Instrument Equipment Co., Ltd., ZRD-3, Beijing, China) was used to measure the cone penetration of the prepared lithium greases.

2.3.2. Tribological Properties

The tribological properties of MoS_2 in grease were investigated by a vertical universal friction and wear testing machine (MMW-1B of Jinan Shunmao Instrument Co., Ltd., Jinan, China). According to the national standard GB-T3142-1982 [23], 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. The GCr15-bearing steel ball has a diameter of 10 mm and a hardness of 62–65 HRC. 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 wear scar diameter was measured by the optical microscope (M203, Aosiwei Optical Instrument Co., Ltd., Shenzhen, China).

2.4. Analysis of Wore Surfaces

The surface morphology of the steel ball was analyzed by scanning electron microscopy (SEM) of Hitachi SU8010. Before the SEM examination, the test balls were ultrasonically cleaned twice with petroleum ether for about 15 min each time. X-ray photoelectron spectroscopy (XPS, Thermo Fisher Scientific K-Alpha, Waltham, MA, USA) is a highly sensitive ultramicroscopic surface analysis technology; it can be used to achieve a 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 Ka rays (hv = 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 test passing-energy 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 MoS_2 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 $\times$ 15 $\times$ 3 mm 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. The corrosion surface of GCr15 bearing steel sheet was evaluated via scanning electron microscopy (SEM) using a Hitachi SU8010, and three-dimensional (3D) morphology was performed using equipment from Zygo Corporation (ZGP OPTICAL PROFILER).

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 MoS_2 additive with different mass fractions in different ways. It can be seen from the data in Table 1 that after adding MoS_2 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 MoS_2 was added, the drop point and cone penetration of the grease changed, indicating that the addition of MoS_2 may have a certain effect on the colloidal structure of lithium grease [24].

Table 1. Dropping point and cone penetration of greases produced by different additional methods of MoS_2 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. Tribological Properties

3.2.1. 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 MoS_2 greases prepared in different ways were estimated. Figure 2 shows the friction coefficient relationship curve and average friction coefficient of MoS_2 greases with different mass fractions prepared by different addition methods. As can be seen from the friction coefficient curve of Figure 2a, when the content of MoS_2 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 cannot play the role of lubrication for a long time during the friction process. The friction coefficient curve corresponding to 0.05 wt% MoS_2 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, which decreases by 19.6% compared with the blank group. The results showed that 0.05 wt% was the best content when MoS_2 was added by "Method A".

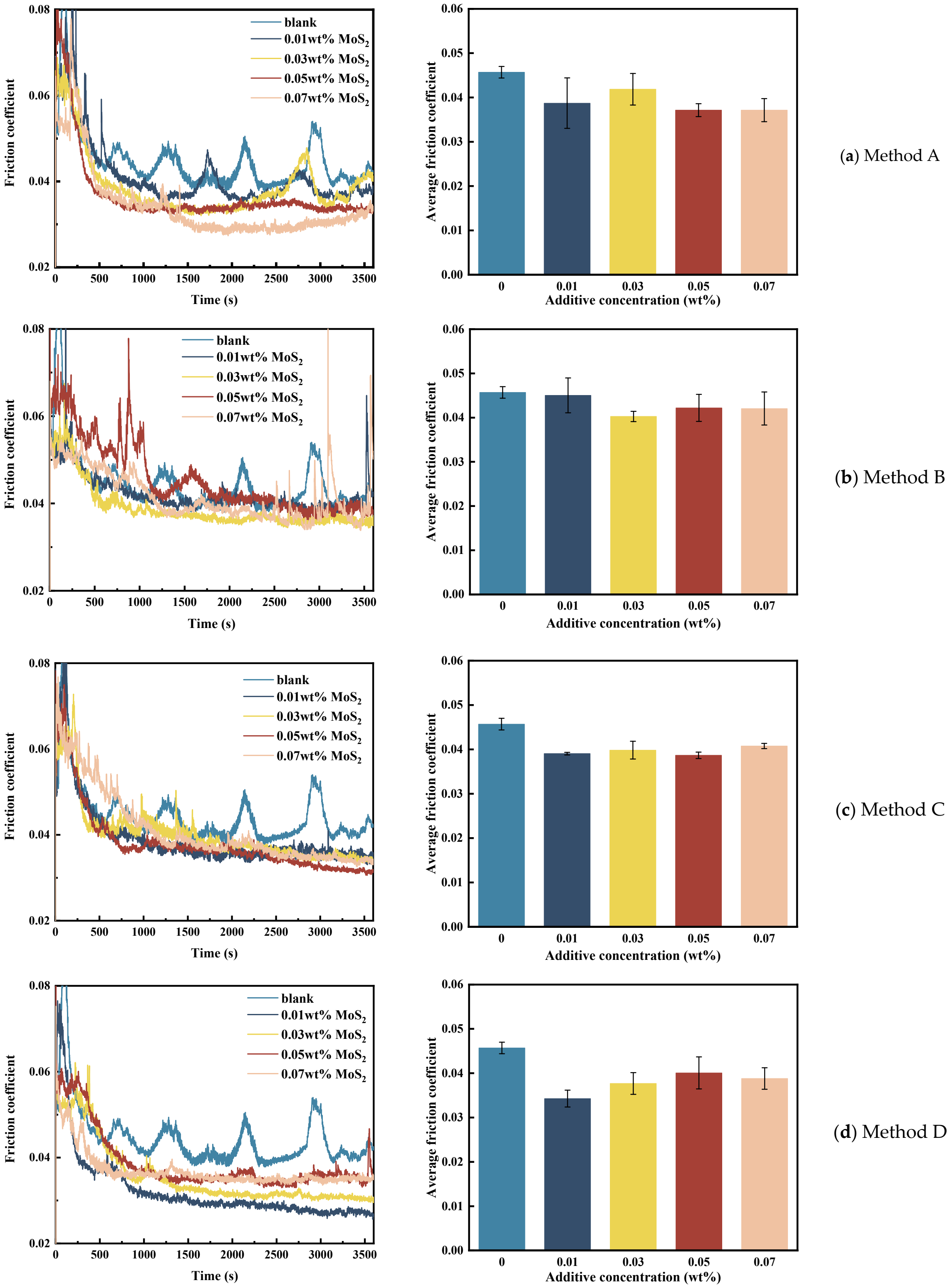
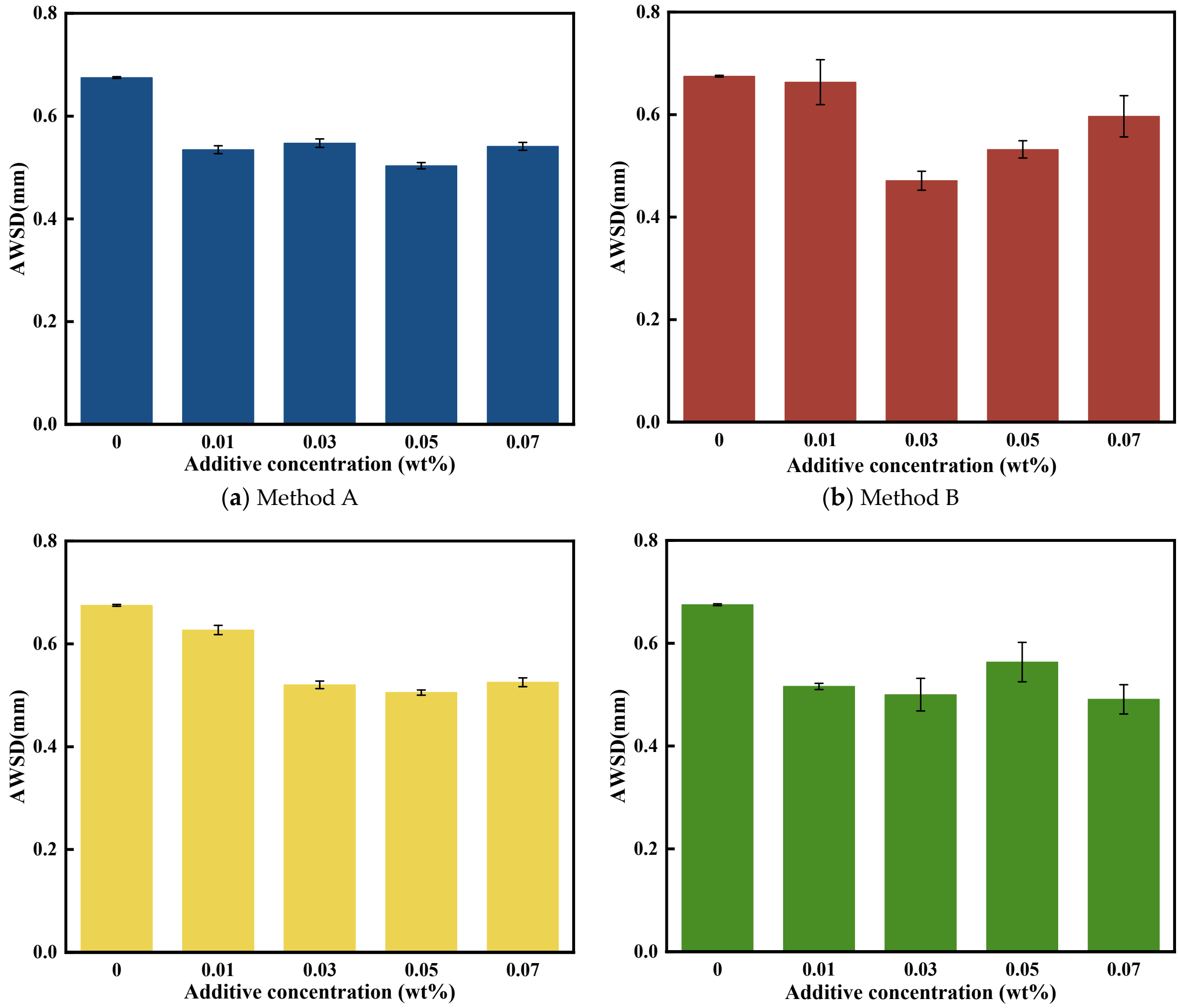


Figure 2. Friction coefficient curves and average friction coefficient of MoS_2 greases prepared by different addition methods with variable amounts of MoS. (392 N, 1450 rpm, 60 min).



(**a**) Method A (**b**) Method B

(**c**) Method C (**d**) Method D

Figure 3. Wear scar diameter of MoS_2 greases prepared by different addition methods with variable amounts of MoS_2. (392 N, 1450 rpm, 60 min).

Figure 2b shows that when MoS_2 was added via "Method B", the average friction coefficient of the prepared grease under experimental conditions first decreased and then increased. By comparing the friction coefficient curves, it can be seen that the optimal addition amount of MoS_2 additive in the grease prepared using this method was 0.03 wt%. The average friction coefficient of MoS_2 grease at the optimal concentration is 0.040, which decreases by 13.0%.

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 in Figure 2c that the friction coefficient of MoS_2 grease prepared via "Method C" was generally stable under experimental conditions, and gradually decreased over time. The three-roll grinding process in "Method C" may help the MoS_2 to be more evenly distributed in the grease, thereby reducing friction for longer. By comparing, it can be found that the optimal MoS_2 additive content of MoS_2 grease prepared via "Method C" was 0.05 wt%, and the corresponding average friction coefficient was 0.039. When compared with the average friction coefficient of 0.046 in the blank group, it can be found that the average friction coefficient was reduced by 15.2%.

"Method C" and "Method D" are different in the addition time of additives. It can be seen in Figure 2d that when MoS_2 was added via "Method D", the friction coefficient curves of MoS_2 grease with different mass fractions were stable, and the optimal content of MoS_2 corresponding to this method was 0.01 wt%, and its average friction coefficient was 0.034. This was a 26.1% reduction compared to the blank group.

Compared with four different MoS_2 adding methods, it can be found that the friction coefficient of grease obtained via "Method D" was the most stable, the average friction coefficient was reduced the most, and the required optimal MoS_2 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 0.01 wt%.

3.2.2. Anti-Wear Performance

Figure 3 shows the average wear scar diameter of MoS_2 greases with different mass fractions prepared by different addition methods under the test conditions of 392 N and 1450 rpm. In Figure 3a, it can be seen that the average wear scar diameter corresponding to the lubricating grease prepared via "Method A" shows that the optimal MoS_2 content was 0.05 wt%, at which point the minimum average wear scar diameter was 0.50 mm. In Figure 3b, it can be seen that the wear scar diameter caused by MoS_2 lubricating grease prepared via "Method B" shows a trend of first decreasing and then increasing with the increase of MoS_2 content, and the optimal content of additives required for the lubricating grease prepared via this method was 0.03 wt%. From the average wear scar diameter in Figure 3c, it can be observed that the optimal amount of MoS_2 added to the lubricating grease prepared via "Method C" was also 0.05 wt%. When the MoS_2 content was 0.01 wt%, a small amount of MoS_2 could not effectively resist wear during the friction process. When the amount of MoS_2 exceeded 0.03 wt%, due to the low surface tension and hydrophilic properties of the MoS_2 sliding layer [25], MoS_2 particles may be unevenly dispersed in the lubricating grease, resulting in a gradual increase in wear scar diameter. Figure 3d shows the corresponding wear scar diameter of the lubricating grease prepared via "Method D". From Figure 3d, it can be seen that the lubricating grease configured with 0.01 wt% MoS_2 exhibits the smallest average wear scar diameter, which was reduced by 0.16 mm compared to the blank group without MoS_2.

3.3. SEM Analysis of Worn Surfaces

Figure 4 shows the SEM images of worn surfaces corresponding to blank lubricating grease and lubricating grease with the optimal mass fraction of MoS_2 added in different ways. In Figure 4a, it can be seen that the worn surface corresponding to the blank grease exhibits obvious wear scar tracks, with wide and large furrows on the wear scar tracks. The wear scar tracks on the steel ball surface (Figure 4b–e) corresponding to the lubricating grease containing the MoS_2 additive become relatively less obvious, and the surface becomes smoother. This may be because MoS_2 has a layered structure that is easy to slide, so it can form a stable film on the friction surface under friction conditions, thereby achieving an anti-wear effect.

By comparing Figure 4b–e, it can be found that the wear scar tracks (300 µm) of "Method A" are relatively uniform. The wear scar tracks (300 µm) of "Method B" are concentrated. "Method C" and "Method D" have a similar range of wear scar tracks. The wear scar tracks of steel balls become shallower in turn, and the MoS_2 grease prepared via "Method D" shows the shallowest wear scar tracks 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 MoS_2 before thickening allows MoS_2 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 MoS_2 to be more evenly distributed in the lubricating grease.

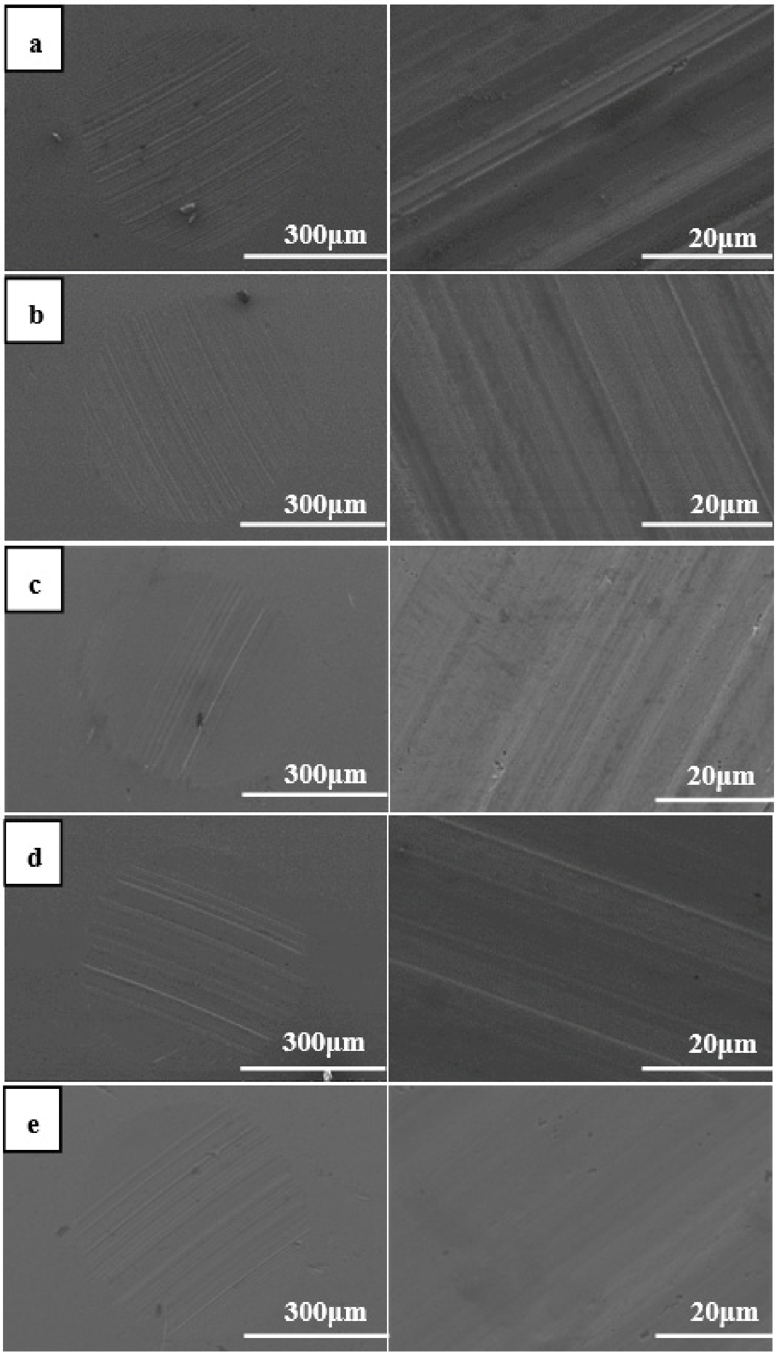


Figure 4. SEM images of worn surfaces: (**a**) blank group, (**b**) Method A with 0.05 wt% MoS_2, (**c**) Method B with 0.03 wt% MoS_2, (**d**) Method C with 0.05 wt% MoS_2, (**e**) Method D with 0.01 wt% MoS_2. (392 N, 1450 rpm, 60 min).

3.4. Soap Fiber Structure of Lithium Lipids

Figure 5 shows the SEM images of the soap fiber structure of blank grease and lithium-based lubricating grease prepared by adding MoS_2 through "Method D" with the best tribological performance 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 [26]. 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. The soap fiber structure of the blank grease and the grease of "Method D" did not change between pre-friction and post-friction, respectively. This shows that the performance of lithium-base grease is excellent, and the addition of MoS_2 has no obvious effect on the structure of soap fiber.

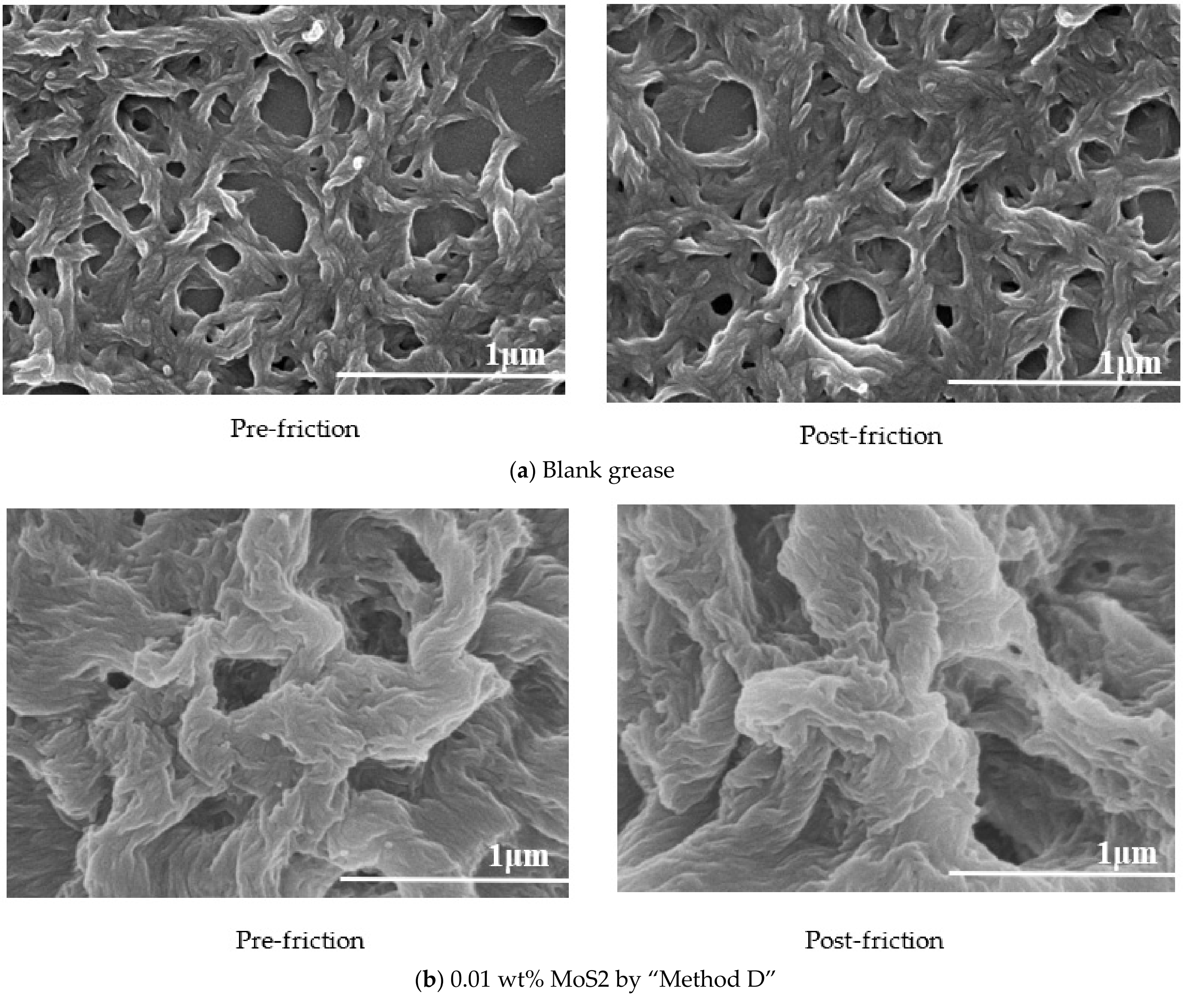


Figure 5. SEM image of soap fiber structure of blank grease and lithium grease with 0.01 wt% MoS_2 via "Method D" before and after friction. (392 N, 1450 rpm, 60 min).

3.5. XPS Analysis of Worn Surfaces

In order to reveal the lubrication mechanism of MoS_2 as an additive in lithium grease, the chemical state of elements on the surface of the wear scar was analyzed by XPS. Figure 6 shows the XPS spectra of C1s, O1s, Fe2p, S2p, and Mo3d on the worn surface of steel balls caused by lithium-based grease with 0.01% MoS_2.

The peaks with binding energies of 284.8, 284.98 and 287.18 eV in the C1s spectra (Figure 6b) correspond to C-C, C-O-C [27] and C=O [28] bonds, respectively. For the O1s (Figure 6c) spectrogram, two photoelectron contribution are found at 529.37 eV and 531.81 eV, attributing to metal oxides (Fe_2O_3) and C-O bonds [29]. The binding energies of Fe2p (Figure 6d) at 706.30 and 711.76 eV peaks correspond to FeS_2 and Fe_2O_3, respectively [30]. The 709.64 and 723.19 eV correspond to Fe $2p_{3/2}$ and Fe $2p_{1/2}$ of FeO. As for the S2p-XPS spectra (Figure 6e), two peaks belonging to MoS_2 [31]/FeS_2 and metal sulfate are found at 161.50 eV and 168.60 eV. The peaks at 232.68 and 235.83 eV in the Mo3d-XPS spectra (Figure 6f) correspond to MoO_2 and MoO_3, respectively [32]. 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 have partially caused tribochemical reactions during the friction process.

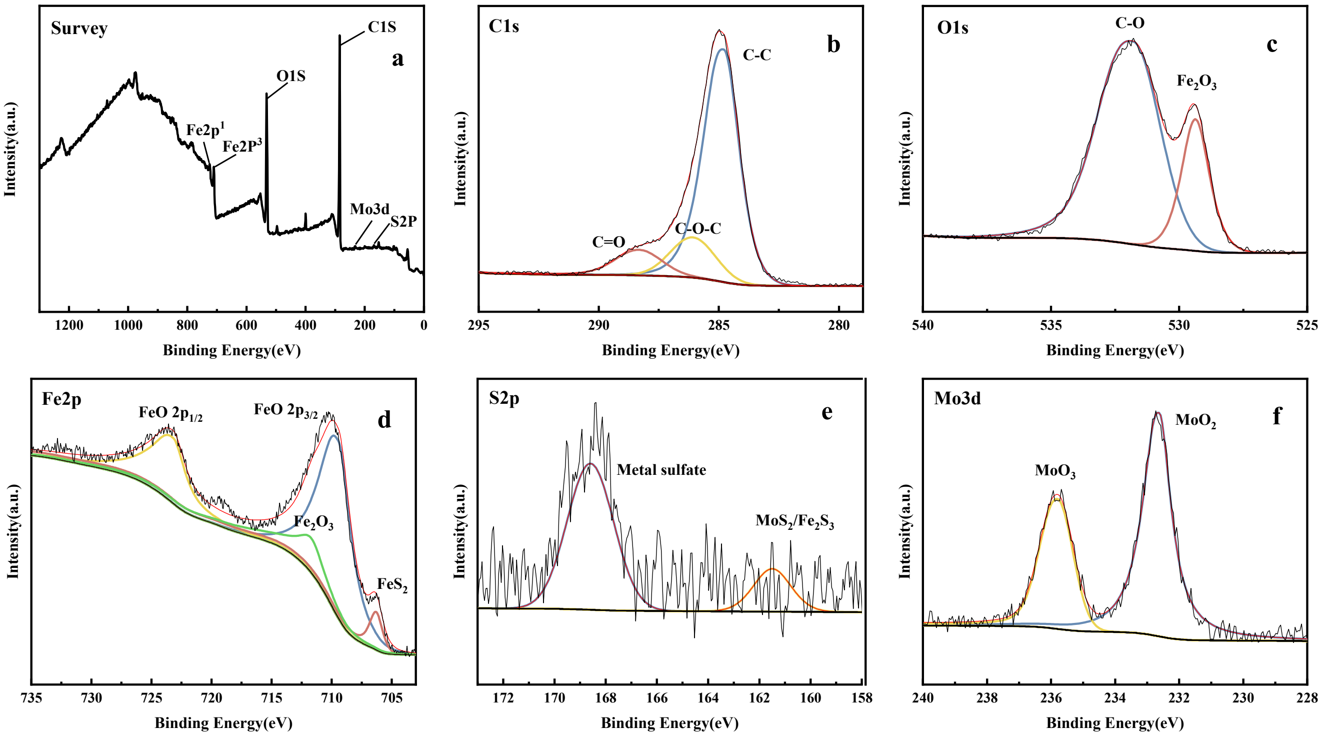


Figure 6. XPS spectra of the worn surface caused by 0.01 wt% MoS_2 lithium grease prepared via "Method D", (**a**) XPS survey spectrum, (**b**) C 1s, (**c**) O 1s, (**d**) Fe 2p, (**e**) S 2p, (**f**) Mo 3d.

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 [33]:

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

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

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 7 shows the corrosion rate of GCr15−bearing steel sheets buried in MoS_2 lubricating grease with different mass fractions prepared by different addition methods for 15 days. Compared with the blank group without MoS_2, it can be found that the addition of MoS_2 additive reduces the corrosion rate of bearing steel in the natural environment, indicating that the addition of MoS_2 effectively slows down the corrosion process of bearing steel.

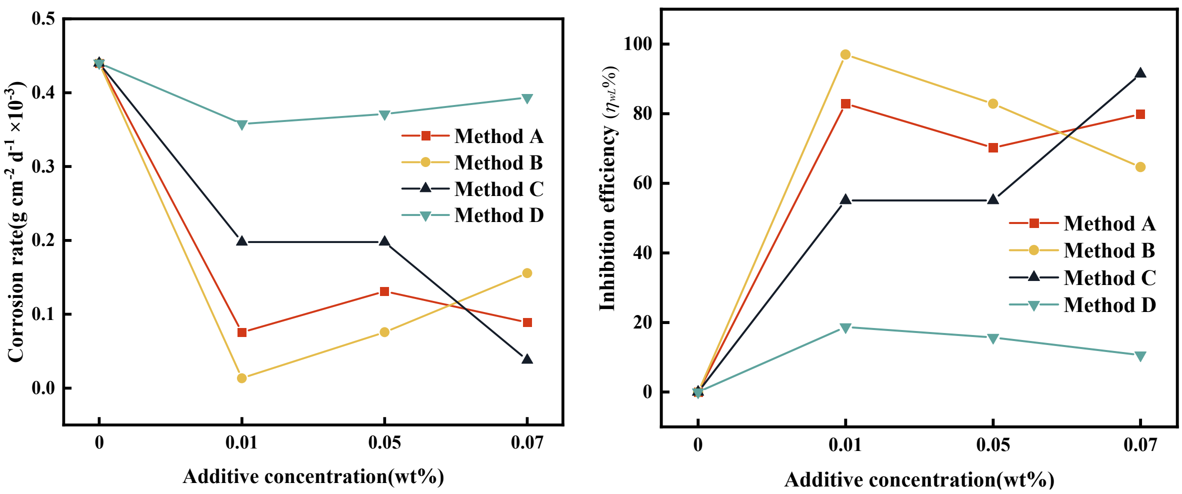


Figure 7. Corrosion rate and corrosion inhibition efficiency of GCr15−bearing steel plate buried in MoS_2 greases with different mass fractions prepared by varying addition methods.

However, as the addition of additives changes, the content of MoS_2 with the best corrosion inhibition efficiency also changes. The optimal corrosion inhibition efficiency of MoS_2 via "Method A" was found at 0.01 wt% MoS_2 content. When MoS_2 was added via "Method B", the optimal corrosion inhibition efficiency occurred when the MoS_2 content was 0.01 wt%. When MoS_2 was added via "Method C", the optimal additive content for corrosion inhibition efficiency was 0.07 wt%. When "Method D" was adopted to add MoS_2, 0.03 wt% MoS_2 exhibited the best corrosion inhibition efficiency. This change may be due to the varying degree of uniform distribution of MoS_2 in the lubricating grease when added in different ways, resulting in different corrosion results. The data in Table 2 and Figure 7 both show that when "Method B" was used to add 0.01 wt% MoS_2, 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%.

The SEM image of the GCr15−bearing steel sheet embedded in 0.01% MoS_2 lubricating grease for 15 days is shown in Figure 8. Figure 8a 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 MoS_2 showed obvious corrosion, with an unsmooth surface and severe corrosion pits (Figure 8b). Figure 8c is the SEM image of the surface of the steel sheet caused by 0.01 wt% MoS_2 grease prepared via "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 0.01 wt% MoS_2 was added via "Method B", the resulting grease causes the slightest corrosion on the surface of the steel sheet (Figure 8d). When 0.01 wt% MoS_2 grease was prepared via "Method C", the corrosion test results in uneven corrosion on the surface of the steel sheet, as shown in Figure 8e. As can be seen from Figure 8f, the grease prepared by adding MoS_2 additive via the process of "Method D" will cause local corrosion on the surface of the steel sheet. From Figure 8c–f, it can be found that MoS_2 grease has a certain corrosion effect on the steel surface, which may be because the S element in MoS_2 can combine with the Fe element in the bearing steel to produce iron sulfide and other products [14], so MoS_2 has a certain corrosion effect, and the surface of the steel sheet was therefore pitted. However, by comparing Figures 8b and 8c–f, it can be found that after the addition of MoS_2 additive, the corrosion phenomenon on the surface of the bearing steel was slowed down in different forms, indicating that MoS_2 has a certain corrosion inhibition effect.

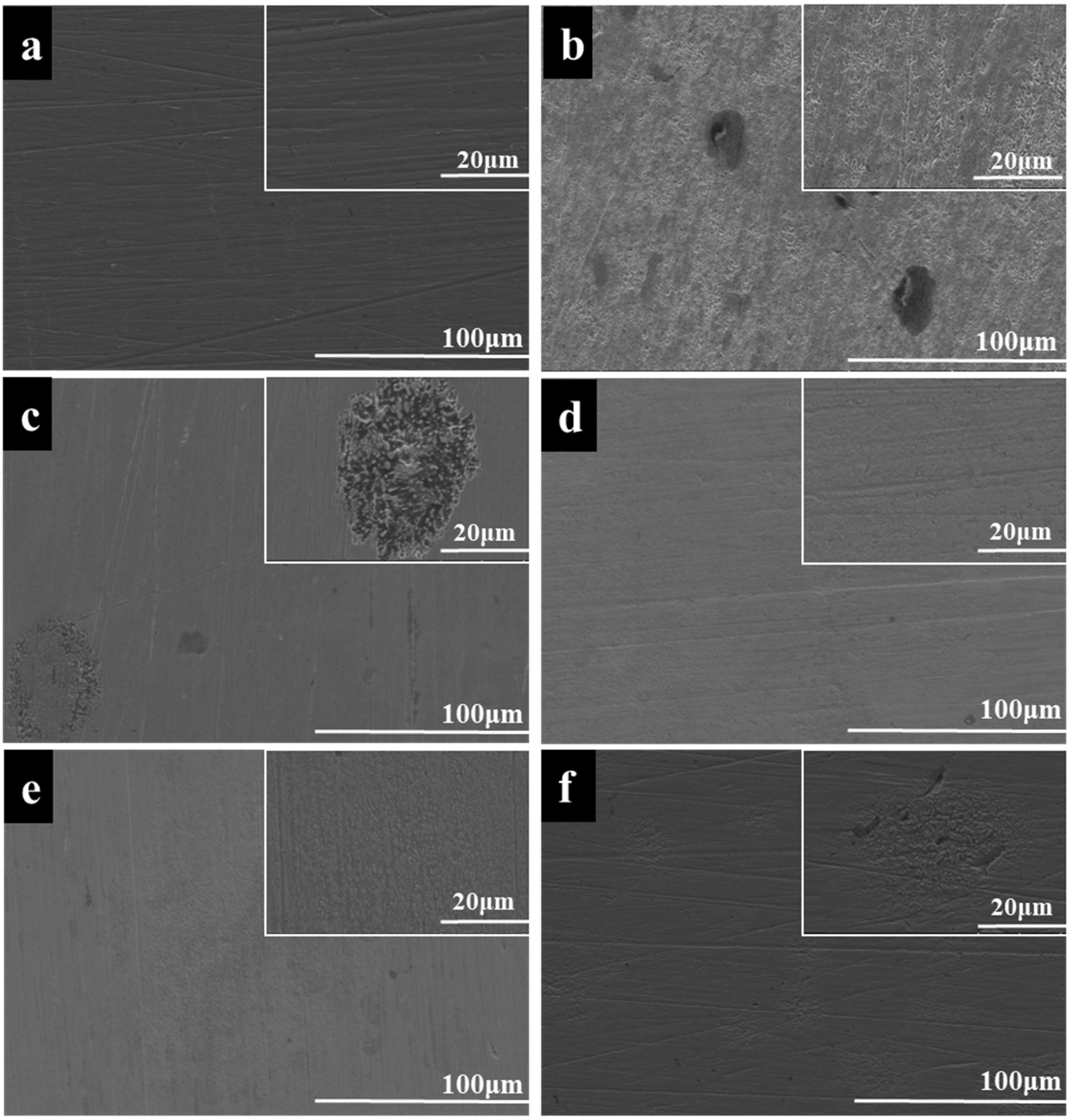


Figure 8. SEM image of GCr15 steel sheet embedded with 0.01 wt% MoS_2 lubricating grease prepared via different addition methods after 15 days: (**a**) original steel surface, (**b**) blank grease, (**c**) Method A, (**d**) Method B, (**e**) Method C, (**f**) Method D.

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 9 shows the 3D morphology of the steel sheet after being embedded in 0.01% MoS_2 grease for 15 days.

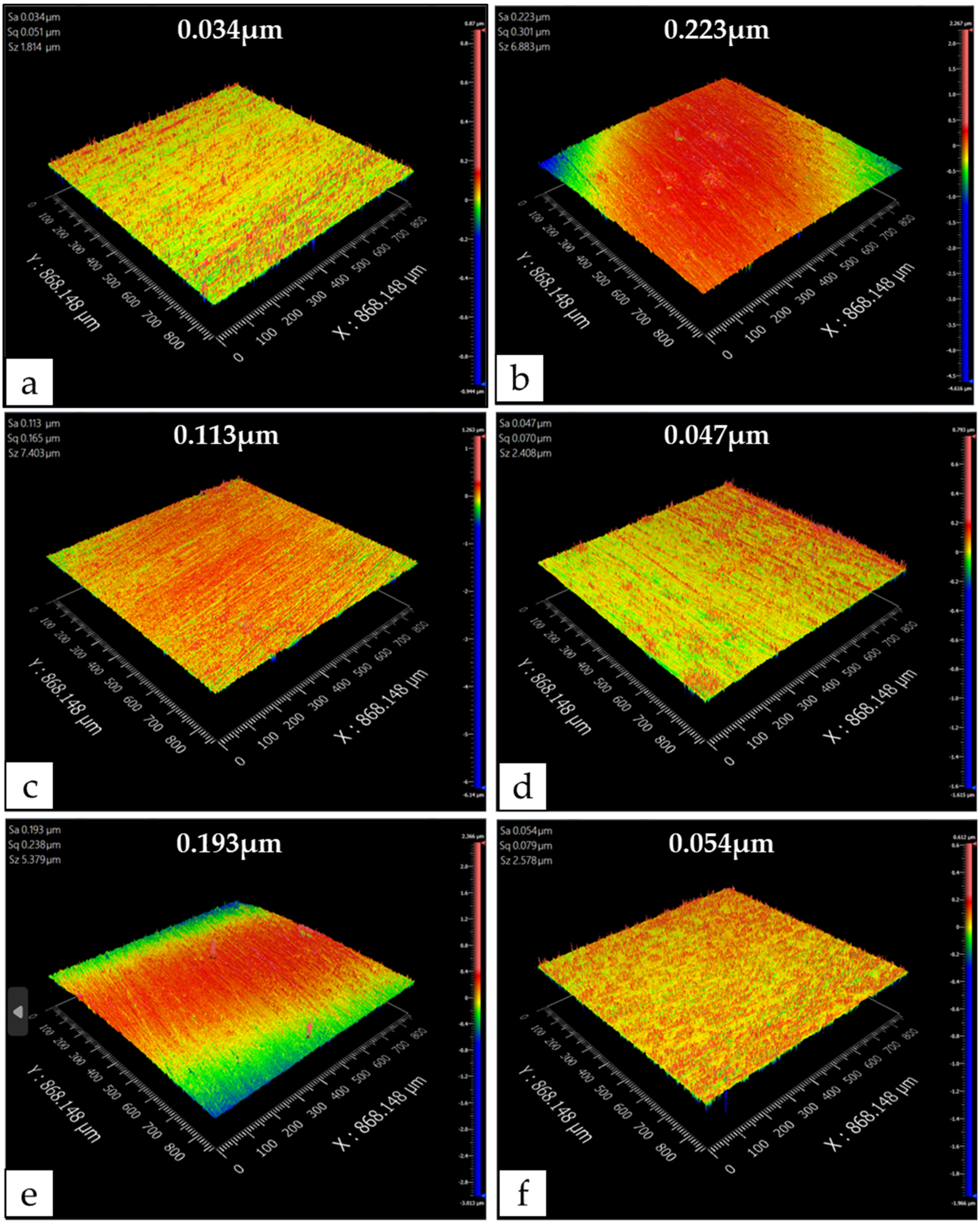


Figure 9. 3D morphology of GCr15−bearing steel sheet embedded in 0.01 wt% MoS_2 lubricating grease prepared via different methods for 15 days: (**a**) original steel sheet, (**b**) blank lubricating grease without MoS_2, (**c**) Method A, (**d**) Method B, (**e**) Method C, (**f**) Method D.

In Figure 9a, 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 MoS_2, the surface roughness of the bearing steel was the highest, reaching 0.223 μm (Figure 9b). Figure 9c illustrates that the surface roughness of the corroded steel plate corresponding to the lubricating grease prepared via "Method A" was 0.113 μm. Figure 9d shows that the roughness of the corroded steel surface corresponding to the lubricating grease prepared via "Method B" was 0.047 μm. Figure 9e,f represent the 3D images of the corroded steel surface corresponding to the lubricating grease prepared via "Method C" and "Method D", respectively, with a surface roughness of 0.193 μm and 0.054 μm.

Comparing Figures 9b and 9c–f, it can be found that regardless of the method of adding 0.01 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 9d) by 0.176 μm. The process of "Method C" (Figure 9e) having the least reduction in surface roughness resulted in a reduction of only 0.03 μm. The 3D morphology in Figure 9 is in good agreement with the SEM results in Figure 8. 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.

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 wear scar diameter was reduced by 0.16 mm. "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. In addition, MoS_2 is oxidized to MoO_2 and MoO_3 during the friction process. The tribochemical reaction occurred between MoS_2 and the rubbing pair, forming a thin friction film, which reduces friction and wear;
4. MoS_2 lithium grease has good anti-corrosion properties, which maybe "Method B" can evenly distribute MoS_2 on the surface of the grease, so that the protective film formed by MoS_2 and GCr15−bearing steel can effectively slow down the corrosion effect of lithium-based grease on steel.

Author Contributions: Writing—original draft, C.Z.; Writing—review & editing, C.Z., Z.H. and Y.W.; Investigation, C.Z.; Funding acquisition, Z.H. and L.X.; Resources, Z.H. and L.X.; Project administration, Z.H.; Supervision, J.L.; Formal analysis, L.L. All authors have read and agreed to the published version of the manuscript.

Funding: 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).

Data Availability Statement: Data are contained within the article.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Meijer, R.J.; Lugt, P.M. The Grease Worker and Its Applicability to Study Mechanical Aging of Lubricating Greases for Rolling Bearings. *Tribol. Trans.* **2021**, *65*, 32–45. [CrossRef]
2. Gurt, A.; Khonsari, M. The Use of Entropy in Modeling the Mechanical Degradation of Grease. *Lubricants* **2019**, *7*, 82. [CrossRef]

3. Wu, C.; Hong, Y.; Ni, J.; Teal, P.D.; Yao, L.; Li, X. Investigation of mixed hBN/Al_2O_3 nanoparticles as additives on grease performance in rolling bearing under limited lubricant supply. *Colloids Surf. A Physicochem. Eng. Asp.* **2023**, *659*, 130811. [CrossRef]
4. Uppar, R.; Dinesha, P.; Kumar, S. A critical review on vegetable oil-based bio-lubricants: Preparation, characterization, and challenges. *Environ. Dev. Sustain.* **2022**, *25*, 9011–9046. [CrossRef]
5. Yadav, A.; Singh, Y.; Negi, P. A review on the characterization of bio based lubricants from vegetable oils and role of nanoparticles as additives. *Mater. Today Proc.* **2021**, *46*, 10513–10517. [CrossRef]
6. Lugt, P.M. Modern advancements in lubricating grease technology. *Tribol. Int.* **2016**, *97*, 467–477. [CrossRef]
7. Wu, L.; Yang, B.; Zhao, F.; Zhang, Z. Tribological Properties of Complex Calcium Sulfonate Grease with Ultrafine SiO_2/MoS_2 Powders. *J. Nano Res.* **2021**, *66*, 35–44. [CrossRef]
8. Chen, X.; Chen, G. Analysis of lithium molybdenum disulfide base grease and its application in roll bearing. *Sci. Technol. Inf.* **2013**, *21*, 119–121. (In Chinese)
9. Wang, Y.; Wu, B. Friction Characteristics and Mechanisms of Two Lithium Greases in Elastohydrodynamic Lubrication. *J. Fail. Anal. Prev.* **2020**, *20*, 1266–1273. [CrossRef]
10. Kanazawa, Y.; Sayles, R.S.; Kadiric, A. Film formation and friction in grease lubricated rolling-sliding non-conformal contacts. *Tribol. Int.* **2017**, *109*, 505–518. [CrossRef]
11. De Laurentis, N.; Cann, P.; Lugt, P.M.; Kadiric, A. The Influence of Base Oil Properties on the Friction Behaviour of Lithium Greases in Rolling/Sliding Concentrated Contacts. *Tribol. Lett.* **2017**, *65*, 128. [CrossRef]
12. Rapoport, L.; Bilik, Y.; Feldman, Y.; Homyonfer, M.; Cohen, S.R.; Tenne, R. Hollow nanoparticles of WS_2 as potential solid-state lubricants. *Nature* **1997**, *387*, 791–793. [CrossRef]
13. Li, J.; Wang, Y.; Liu, D.; He, K.; Lu, L. Research progress on the properties and application of molybdenum disulfide. *Powder Metall. Technol.* **2021**, *39*, 471–478. (In Chinese) [CrossRef]
14. Niste, V.B.; Ratoi, M. Tungsten dichalcogenide lubricant nanoadditives for demanding applications. *Mater. Today Commun.* **2016**, *8*, 1–11. [CrossRef]
15. Wu, Z. Synthesis of Molybdenum (Tungsten) Disulfide Nanostructures and Their Properties. Ph.D. Thesis, Central South University, Changsha, China, 2012.
16. Wen, P.; Lei, Y. Preparation and Tribological Properties of Covalent Organic Framework Nanomaterials. *Tribology* **2022**, *42*, 123–130. (In Chinese) [CrossRef]
17. Cheng, S.; Guo, X.; Jiang, M.; He, Y. Research progress of lithium complex grease. *Contemp. Chem. Ind.* **2018**, *47*, 152–158. (In Chinese) [CrossRef]
18. Xie, L.; Lu, Z.; Kong, X.; Chen, L.; Luo, H. Study on the effect of ZnS nano additive on the lubricationperformance of complex calcium-based grease and its mechanism. *J. Baoji Univ. Arts Sci. (Nat. Sci.)* **2023**, *43*, 41–47. (In Chinese) [CrossRef]
19. Cheng, Y. Preparation and Tribological Properities of Space Lubricating Grease Containing MoS_2 Nanoparticles. Master's Thesis, Hefei University of Technology, Hefei, China, 2012.
20. He, Z.; Zhu, X.; Tang, J.; Qiu, W.; Rao, S. Tribological Properties of Nano Lanthanum Oxide as Additives in Lithium Base Greases with Rapeseed Oil. *Chin. Rare Earths* **2017**, *38*, 23–29. (In Chinese) [CrossRef]
21. *GB-T4929-1985*; Lubricating Grease-Determination of Dropping Point. Standards Press of China: Beijing, China, 1985.
22. *GB-T269-1991*; Lubricating Grease and Petrolatum-Determinationof Cone Penetration. Standards Press of China: Beijing, China, 1992.
23. *GB/T 3142-1982*; Lubricants-Determination of Load-Carrying Capacity (Four Balls Method). Standards Press of China: Beijing, China, 1990.
24. Wang, W.; Tian, S.; Sun, H. Influence of base oil and additives on microstructure of grease. *Pet. Prod. Appl. Res.* **2015**, *33*, 26–34. (In Chinese) [CrossRef]
25. Yuan, S. Study on the surface modification and friction properties of molybdenum disulfide. *Mod. Salt Chem. Ind.* **2019**, *46*, 50–54. (In Chinese) [CrossRef]
26. Zhang, E.; Li, W.; Zhao, G.; Wang, Z.; Wang, X. A Study on Microstructure, Friction and Rheology of Four Lithium Greases Formulated with Four Different Base Oils. *Tribol. Lett.* **2021**, *69*, 98. [CrossRef]
27. 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. [CrossRef] [PubMed]
28. Liang, Y.; He, X.; Chen, L.; Zhang, Y. Preparation and characterization of TiO_2–Graphene@Fe_3O_4 magnetic composite and its application in the removal of trace amounts of microcystin-LR. *RSC Adv.* **2014**, *4*, 56883–56891. [CrossRef]
29. 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. [CrossRef]
30. 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 MoS_2 Nanosheets. *Lubricants* **2023**, *11*, 336. [CrossRef]
31. 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. [CrossRef]

32. 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 MoS_2/h-BN nano-additives. *Wear* **2022**, *504–505*, 204426. [CrossRef]
33. 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. *Corros. Sci.* **2021**, *185*, 109414. [CrossRef]