

Article

# Tribological Investigation of Layered Zirconium Phosphate in Anhydrous Calcium Grease

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**Abstract:** The tribological properties of  $\alpha$ -zirconium phosphate particles as an additive in anhydrous calcium grease were studied by using an Optimol SRV-V oscillating reciprocating tester and a four-ball tester. Fortunately,  $\alpha$ -Zr(HPO<sub>4</sub>)·H<sub>2</sub>O ( $\alpha$ -ZrP) grease exhibits excellent properties in anti-friction and wear-resistant, load-carrying capacity, and extreme pressure properties. Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS) and 3D analysis show that  $\alpha$ -ZrP particles appear to form a protective film allowing increased load capacity and operating frequency of the rubbed pairs. Meanwhile,  $\alpha$ -ZrP particles can provide low friction coefficient and wear loss during a long-term test.

**Keywords:** solid lubricants;  $\alpha$ -Zr(HPO<sub>4</sub>)·H<sub>2</sub>O; anhydrous calcium grease

## 1. Introduction

It is reported that a large amount of fuel energy is spent to conquer the mechanical losses because of friction, as in paper machines, passenger cars, trucks, and buses, from a comprehensive study by Holmberg et al. [1–3]. High performance, affordable, and long-term lubricants have been relentlessly explored for the efficiency and durability of equipment operation. Solid lubricants offer alternatives to the lubricant formulator for situations where traditional liquid additives fall short on performance, such as high-temperature, low sliding speeds under high contact stresses on bearing points of machinery [4–7]. Many kinds of solid lubricants have been considered to be promising candidates for lubricant additives in practice [8–10].

Our recent study demonstrated that  $\alpha$ -Zr(HPO<sub>4</sub>)·H<sub>2</sub>O ( $\alpha$ -ZrP) had good performance in anti-wear, friction-reduction, and load-carrying capacity as a solid lubricating additive in lithium grease [11,12]. Whether under rolling mode or reciprocating mode,  $\alpha$ -ZrP has better tribological property than the conventional solid lubricant molybdenum disulfide. Similar to molybdenum disulfide,  $\alpha$ -ZrP is a layered cation exchange material of Zr(HPO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O, whose crystal structure consists of Zr<sup>4+</sup> ions positioned alternately slightly above and below the ab plane, bonded with the oxygen atoms of six different tetrahedral phosphate groups, producing a two-dimensional cross-linked covalent network plane [13]. The weak bond strengths between the planes allow for easy shearing of the crystal, resulting in a lamellar mechanism of lubrication.  $\alpha$ -ZrP particles can form a protective film to prevent the two friction pairs direct contact. These properties make  $\alpha$ -ZrP promising candidates for lubricant additives.

In this study, the tribological properties of  $\alpha$ -ZrP as an additive in anhydrous calcium grease were investigated by using an Optimol SRV-V tester and a four-ball tester. The findings of this study will further expand the application scope of  $\alpha$ -ZrP as a solid lubricant. The anhydrous calcium grease is outstanding in water resistance, colloid stability, corrosion resistance, and salt spray corrosion resistance [14,15]. In order to better understand the tribological properties of  $\alpha$ -ZrP, we selected MoS<sub>2</sub> and graphite as the reference in this research. Because MoS<sub>2</sub> and graphite are the conventional solid

lubricant, which are usually used as additives in calcium grease [16–19]. The tribological performance of  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite anhydrous calcium grease was evaluated at different applied loads, frequencies, temperatures, and durations. A 3D optical profiling system, scanning electron microscopy (SEM), and energy dispersive X-ray spectroscopy (EDS) were employed to study traces of the friction surface to obtain an adequate understanding of the lubrication mechanism.

## 2. Experimental Section

### 2.1. Materials

The chemical reagents were obtained commercially and were used without further treatment. Poly-alpha-olefin PAO8 (viscosity of 46.48 mm<sup>2</sup>/s at 40 °C, viscosity index of 146, Exxon Mobil Corp., Irving, TX, USA), 12-hydroxystearic acid (Development Co., Ltd., Shanghai, China), calcium hydroxide (AR, 95%, Aladdin Industrial Corporation, Shanghai, China), MoS<sub>2</sub> (DAI ZO Corporation, Osaka, Japan), graphite (Aoyu Graphite Factory, Jixi, China), petroleum ether (Tianjin Fengchuan Chemical Reagent Science and Technology Co. Ltd., Tianjin, China). Distilled water was prepared in our laboratory.  $\alpha$ -ZrP was synthesized using the reported method [11].

The laboratory anhydrous calcium-based grease was obtained as follows. The mixture of weighed 12-hydroxystearic acid and PAO8 was first put into the grease kettle and heated to 85 °C until it became homogeneous. Second, a water slurry of calcium hydroxide was added slowly to the above mixture. The mixture was heated to 120 °C for 1.5 h, then the temperature increased to 140 °C for 10 min, the remaining PAO8 was added to the grease kettle, stirred until the temperature cooled to room temperature, and the base anhydrous calcium-based grease was obtained. Varying concentrations (1.0–7.0 wt %) of  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite were added to the calcium grease to investigate the effect of these additives on the tribological behavior of grease. Each calcium grease sample was mixed via mechanical stirring and ground three times in a triple-roller mill.

### 2.2. Tribological Test

The tribological behavior of the  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite anhydrous calcium grease was evaluated by an Optimol SRV-V oscillating friction and wear tester (Optimol instruments Prueftechnik GmbH, Muenchen, Germany). The upper ball made of AISI 52100 steel (diameter 10 mm, HRC 59–62, Ra 0.014  $\mu$ m) slides reciprocally with an amplitude of 1 mm on the stationary AISI 52100 steel discs (diameter 24 mm, 7.85 mm height, HRC 59–62, Ra 0.014  $\mu$ m). The grease volume was applied with grease caliper provided by Optimol instruments (Optimol instruments Prueftechnik GmbH, Muenchen, Germany). Before the test, about 0.2 g of grease was fed into the ball–disc contact area [12]. Before each test, the balls and the disks were cleaned in petroleum ether. We chose different applied loads, frequencies, and temperatures to determine the tribological performance of lubricating grease, and the experimental conditions were: loads 300 to 900 N, frequencies 20 to 70 Hz, temperatures 25 to 80 °C, 1.0 mm stroke, and duration of 30 min.

The friction coefficient curve was recorded automatically via designated configuration of SRV-V tester. It was necessary that every test was repeated three times to receive reliable experimental results. Therefore, the mean friction coefficient and wear volume can be obtained by averaging the data of three experiments.

In addition, the tribological behavior of  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite as additives in calcium grease was also evaluated on the four-ball friction and wear tester (Xiamen Tenkey Co., Ltd., Xiamen, China) according to ASTM D 2596, which was beneficial to obtain a comprehensive understanding of the performance of lubricating grease with three additives.

### 2.3. Characterization

After the test, the lower disc was cleaned with petroleum ether for 5 min in an ultrasonic bath. The wear scar and worn surfaces of the disc were measured by using a 3D optical profiling system

(Zygo, ZeGage, Middlefield, CT, USA) and scanning electron microscopy (HITACHI, TM-3000, Hitachi, Japan). The elemental composition of the worn surface was analyzed by energy dispersive spectrometer (Bruker, QUANTAX 70, Billerica, MA, USA).

### 3. Results and Discussion

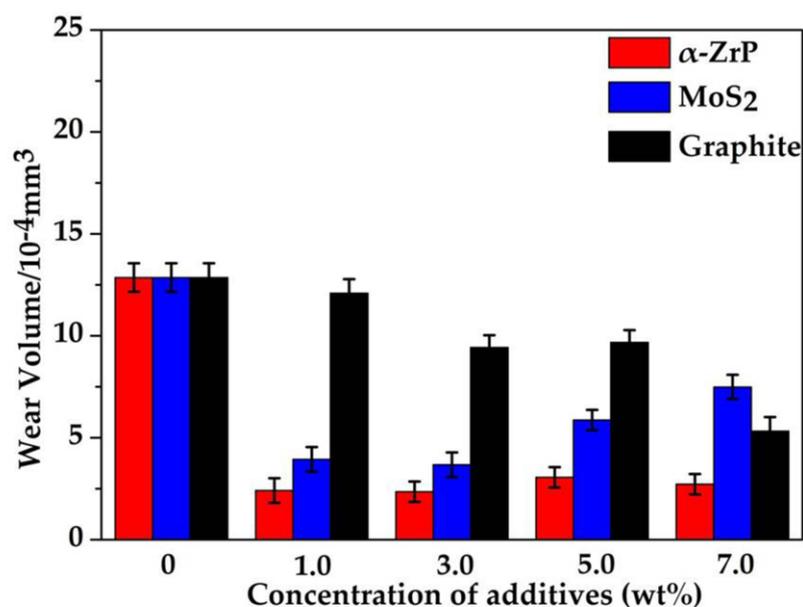
#### 3.1. Determination of Suitable Concentration of Additives

The suitable concentration of the three additives in anhydrous calcium grease depended on the evaluation of wear volume losses of the steel discs. The concentration of additives ranged from 1.0 to 7.0 wt %. The results for each additive differed depending on additive concentration. From Figure 1, it can be seen that the anti-wear capacities of base grease were improved by the addition of  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite, respectively. The addition of  $\alpha$ -ZrP led to the lowest wear volume of the steel discs in the test range. For MoS<sub>2</sub>, the wear volume had a significant increase when the concentration reached 5.0 wt %. For graphite, the wear volume declined gradually with the increase of the concentration. According to the results of the above experiment, the optimized concentration is based on having effective lubrication at the lower concentration. Considering that the friction test should be under the same condition, the feasible concentration for the three additives in anhydrous calcium grease was chosen to be 3.0 wt %. Typical properties are listed in Table 1.

**Table 1.** Typical properties of pure anhydrous calcium grease and the grease containing 3.0 wt % additives.  $\alpha$ -ZrP:  $\alpha$ -Zr(HPO<sub>4</sub>)·H<sub>2</sub>O.

Grease Property	Test Standard	Base Grease	$\alpha$ -ZrP	MoS <sub>2</sub>	Graphite
Dropping point (°C)	ASTM D 566	159	153	158	160
Cone penetration (0.1 mm)	ASTM D 217	315	310	303	309
Wear scar diameter (WSD/mm) <sup>1</sup>	ASTM D 2266	0.45	0.34	0.51	0.43
Maximum non-seizure load ( $P_B/N$ ) <sup>2</sup>	ASTM D 2596	549	1236	696	618
Welded point ( $P_D/N$ ) <sup>3</sup>	ASTM D 2596	1569	1961	1961	1569

<sup>1</sup> Experimental condition: four-ball test, 294 N, 1450 r/min, 30 min, 75 °C; <sup>2,3</sup> Experimental condition: four-ball test, 1770 r/min, 10 s, room temperature.

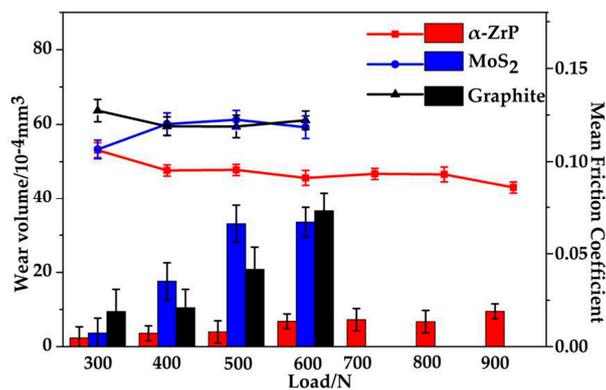


**Figure 1.** The wear volume losses of the steel discs as a function of concentration for anhydrous calcium greases with  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite (SRV test, load 300 N, frequency 30 Hz, temperature 80 °C, duration 30 min).

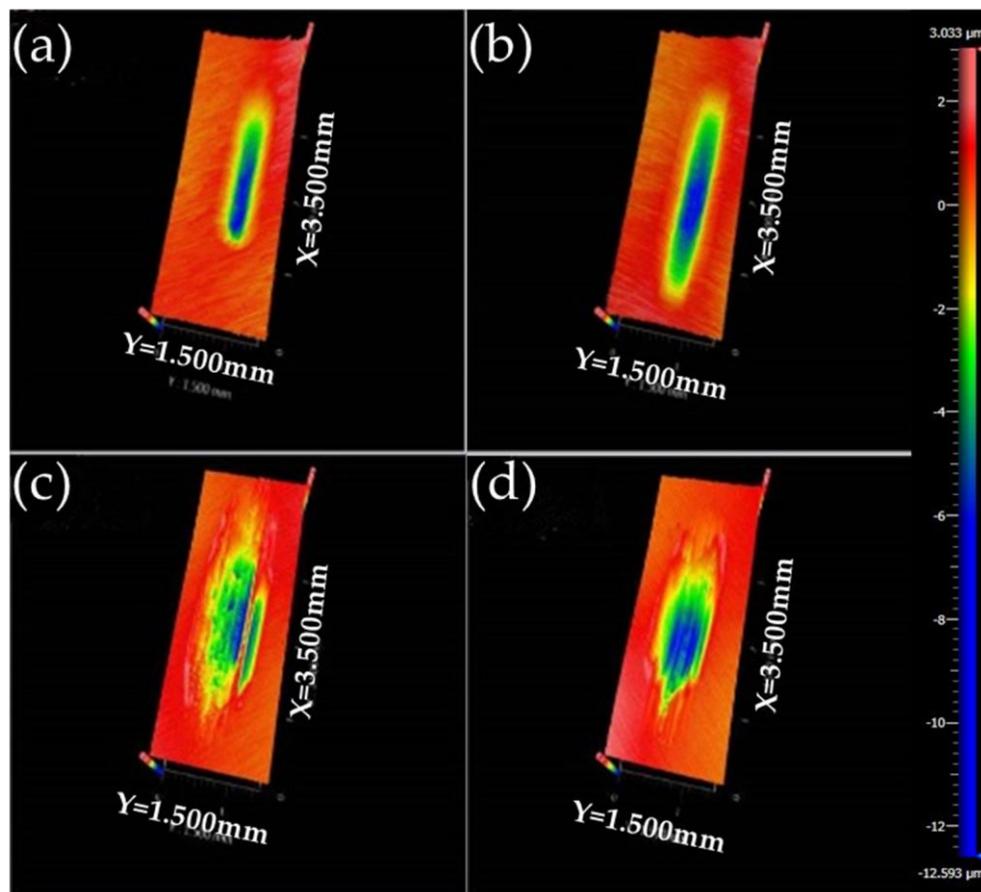
### 3.2. The Effect of Different Loads on the Performance of Grease

The maximum non-seizure load ( $P_B$  value) and welded load ( $P_D$  value) of the additives were first tested to evaluate their extreme pressure (EP) property with a four-ball tester (Table 1). It is worth mentioning that the duration of the EP measurement was only ten seconds, which is mainly determined by the film formation velocity and the mechanical properties of the adsorption film on the rubbing surface. Table 1 shows the effect of the additives on  $P_B$  value and  $P_D$  value. Comparing these three additives, the  $P_B$  value of  $\alpha$ -ZrP was 1236 N, which is 2.3 times that of the base grease. For  $\text{MoS}_2$ , the maximum  $P_B$  value was 696 N, while the maximum  $P_B$  value of graphite was 618 N. The  $\alpha$ -ZrP and  $\text{MoS}_2$  grease had the same  $P_D$  value, which is 1961 N (1.2 times that of the base grease). For graphite, the welded load was 1569 N, which is the same  $P_D$  value as the base grease. Apparently,  $\alpha$ -ZrP had the best extreme pressure property among the three additives, which indicates that  $\alpha$ -ZrP might adhere more easily onto the metal surface to prevent the metal surfaces from seizing each other.

In order to better understand the tribological performance of the three additives based on anhydrous calcium greases, the Optimol SRV-V tester was employed to investigate the variation in mean friction coefficient and wear volumes of the steel discs under the applied loads from 300 to 900 N, which corresponds to a Hertz contact pressure from 3.138 to 4.526 GPa, respectively. Figure 2 compared  $\alpha$ -ZrP grease with  $\text{MoS}_2$  and graphite grease; the carrying applied load of  $\alpha$ -ZrP grease reached 900 N, the mean friction coefficient was steady throughout the test, the wear volumes varied slightly with the increase of the applied load, the mean friction coefficient and wear volume of  $\alpha$ -ZrP were the lowest among the three additives. For  $\text{MoS}_2$  and graphite grease, the maximum applied load was 600 N. The variation trend of friction coefficient for  $\text{MoS}_2$  was almost same as that of graphite, but the wear volumes of graphite were lower than that of  $\text{MoS}_2$  as the applied load increased. The 3D microscopic images of the corresponding wear scars on the steel discs are provided in Figure 3. For  $\alpha$ -ZrP grease, circular and smooth wear surfaces were observed at relatively low wear loss. Deep furrows along the motion direction were displayed on the worn surface of  $\text{MoS}_2$  and graphite grease. It can be seen that  $\alpha$ -ZrP possesses better anti-friction and anti-wear properties than  $\text{MoS}_2$  and graphite under higher applied load.



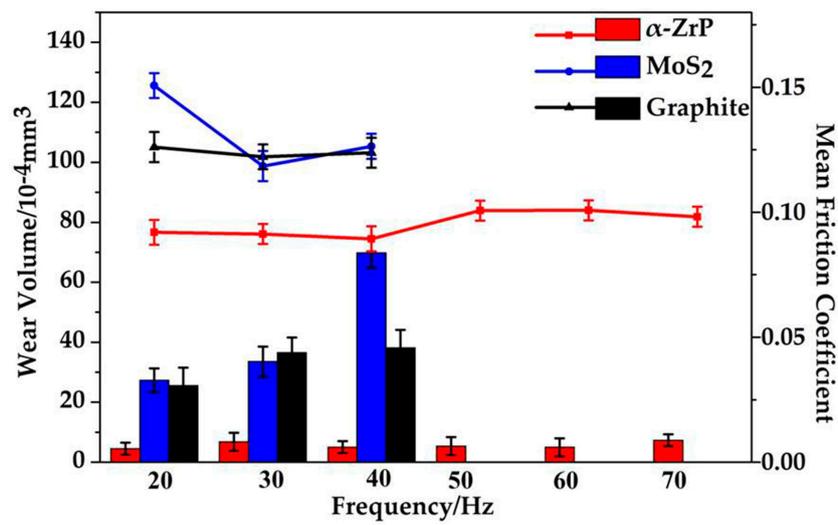
**Figure 2.** The wear volume (columns) and the mean friction coefficient (lines) for different applied loads for the grease with  $\alpha$ -ZrP,  $\text{MoS}_2$ , and graphite (SRV test, frequency 30 Hz, temperature 80 °C, duration 30 min).



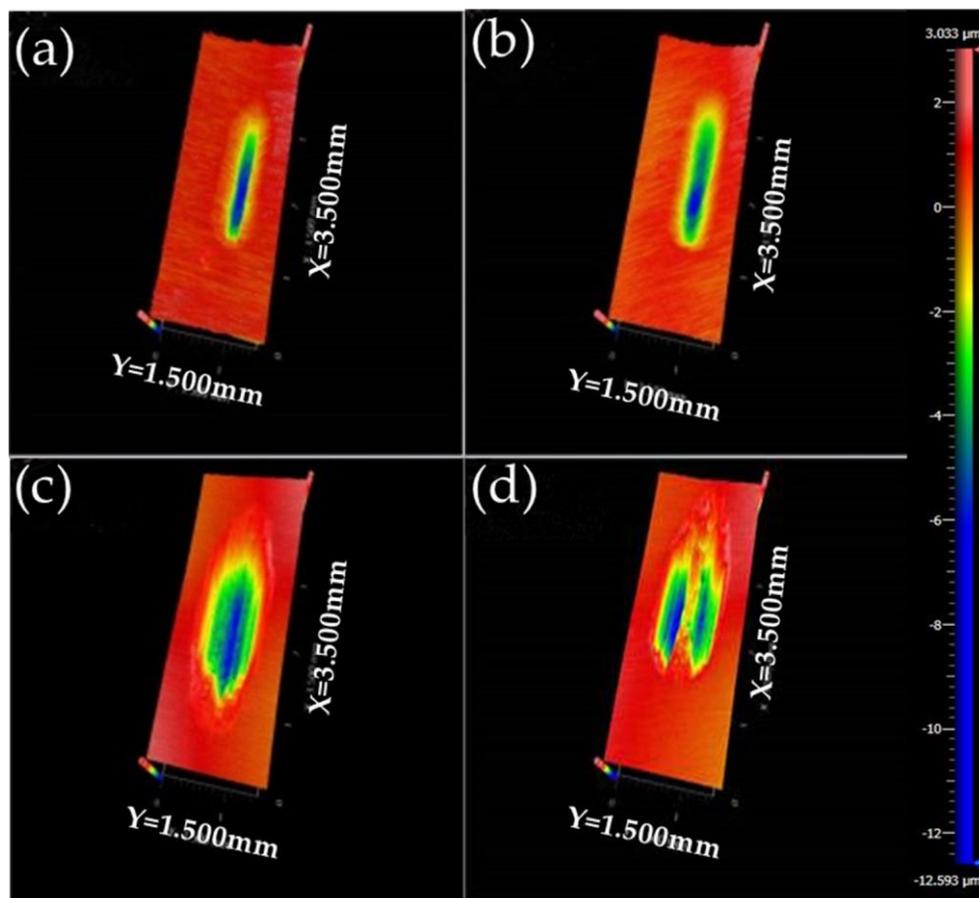
**Figure 3.** 3D microscopic images of wear tracks on the lower disks: (a)  $\alpha$ -ZrP at 600 N; (b)  $\alpha$ -ZrP at 900 N; (c) MoS<sub>2</sub> at 600 N; (d) graphite at 600 N (SRV test, frequency 30 Hz, temperature 80 °C, duration 30 min).

### 3.3. The Effect of Different Frequencies on the Performance of Grease

According to the above the applied load test result, the applied load was chosen to be 600 N under the experimental frequency from 20 to 70 Hz. Figure 4 gives the friction and wear behavior as a function of frequency with the three additives by using the SRV tester. It can be seen that the maximum operating frequency for MoS<sub>2</sub> and graphite was only 40 Hz, whereas it was 70 Hz for  $\alpha$ -ZrP. Compared to the other two greases, the parameters of wear volume and average friction coefficient for  $\alpha$ -ZrP still remained at the lowest level under the experimental conditions. At the running frequency of 40 Hz, the wear volumes ( $10^{-4}$  mm<sup>3</sup>) for  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite grease were  $5.02 \pm 2.15$ ,  $69.84 \pm 5.08$ , and  $38.09 \pm 6.13$ , respectively. The corresponding average friction coefficients for  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite grease were 0.089, 0.126, and 0.124, respectively. Under the experimental conditions,  $\alpha$ -ZrP grease possessed the best anti-wear and friction-reducing performance among the three additives. Figure 5 presents the 3D surface profiles of the corresponding wear tracks on discs after friction tests. It is obvious that the wear track of the disk lubricated by  $\alpha$ -ZrP remained shallow and narrow with increasing testing frequency, which further confirms the superior lubrication properties of  $\alpha$ -ZrP.



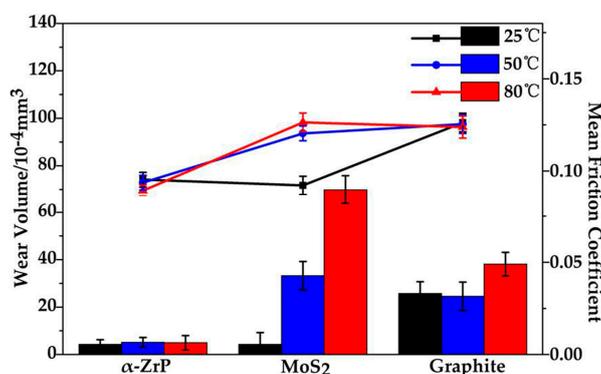
**Figure 4.** The wear volume (columns) and mean friction coefficient (lines) for different frequencies for the grease with  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite (SRV test, load 600 N, temperature 80 °C, duration 30 min).



**Figure 5.** 3D microscopic images of wear tracks on the lower disks: (a)  $\alpha$ -ZrP at 40 Hz; (b)  $\alpha$ -ZrP at 70 Hz; (c) MoS<sub>2</sub> at 40 Hz; (d) graphite at 40 Hz (SRV test, load 600 N, temperature 80 °C, duration 30 min).

### 3.4. The Effect of Different Temperatures on the Performance of Grease

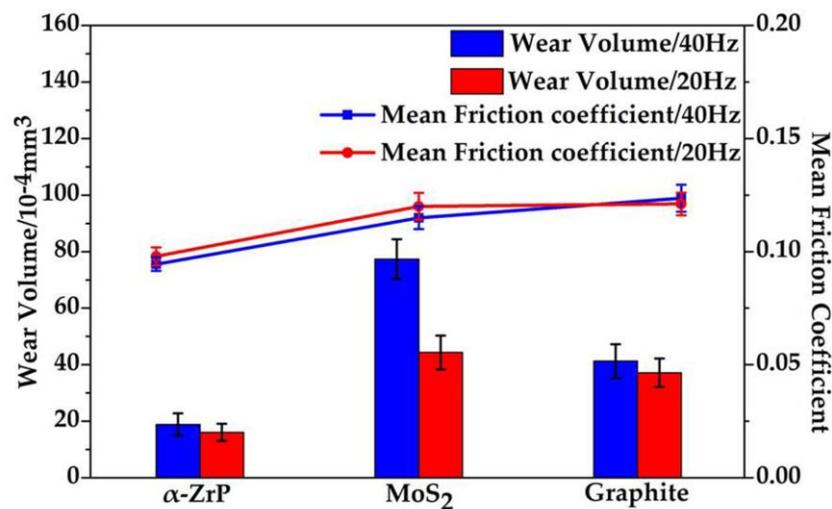
The influence of temperatures on the tribological properties of the three greases was studied in this work. The tests were conducted at 25, 50, or 80 °C. The applied load was 600 N and the frequency was 40 Hz (Figure 6). With increasing temperature from 25 to 80 °C, the wear volumes ( $\times 10^{-4} \text{ mm}^3$ ) for the  $\alpha$ -ZrP grease were  $4.24 \pm 2.05$ ,  $5.18 \pm 2.11$ , and  $5.02 \pm 3.09$ , respectively; for MoS<sub>2</sub> grease, values were  $4.28 \pm 5.25$ ,  $33.19 \pm 6.18$ , and  $69.84 \pm 6.29$ , respectively; for graphite grease, values were  $25.71 \pm 5.21$ ,  $24.53 \pm 6.08$ , and  $38.09 \pm 5.11$ , respectively. The  $\alpha$ -ZrP grease still kept the lowest wear values. The wear volumes of  $\alpha$ -ZrP and graphite grease had a slight dependence on the temperature, while the wear values of MoS<sub>2</sub> grease increased obviously with the rise of the temperature. This may be due to MoS<sub>2</sub> easily oxidized to MoO<sub>3</sub> in higher-temperature environments. The average friction coefficients for  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite exhibited analogous trends. The temperature had little effect on the friction coefficient for both  $\alpha$ -ZrP and graphite grease. Meanwhile, the mean friction coefficient for MoS<sub>2</sub> grease increased from 0.0922 to 0.1264 as the temperature ranged from 25 to 80 °C. Compared with MoS<sub>2</sub> and graphite,  $\alpha$ -ZrP particles presented good anti-friction and wear resistant properties; meanwhile, this material also holded stable tribological properties under harsh loads and frequencies, even over a wide temperature range.



**Figure 6.** The wear volume (columns) and mean friction coefficient (lines) for different temperatures for the grease with  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite (SRV test, load 600 N, frequency 40 Hz, duration 30 min).

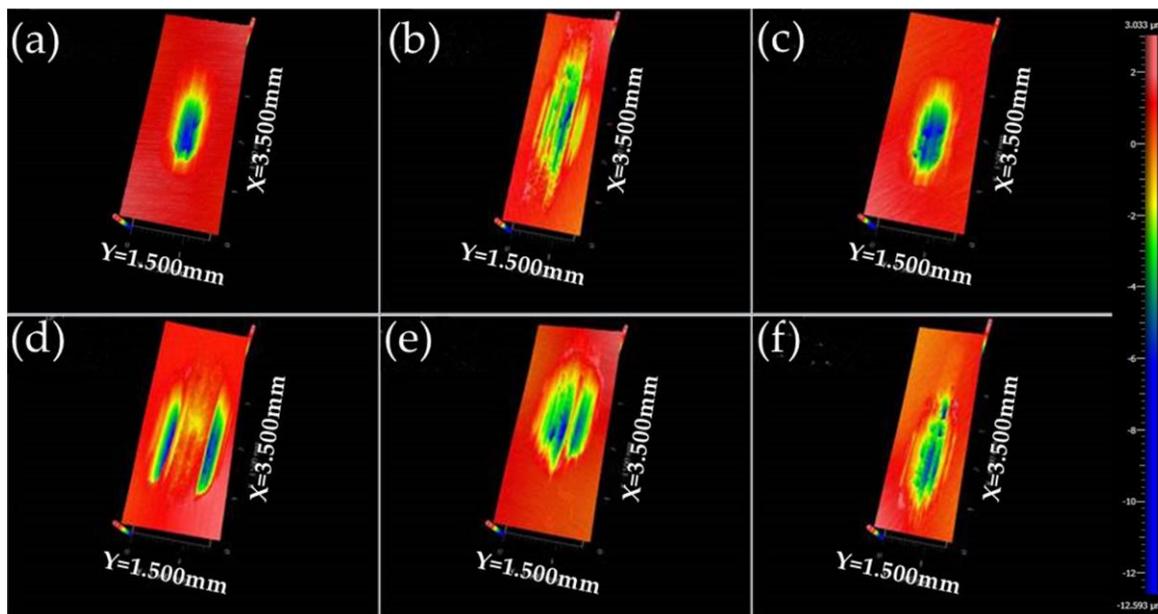
### 3.5. The Effect of Long Durations on the Performance of Grease

Based on actual operating condition of grease, we performed a prolonged test to study the quality of grease with  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite in test conditions of 600 N–40 Hz and 600 N–20 Hz. The test time lasted 360 min. Under the test conditions of 600 N–20 Hz–360 min, the wear volumes ( $10^{-4} \text{ mm}^3$ ) for  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite grease were  $16.10 \pm 2.02$ ,  $44.31 \pm 5.11$ , and  $37.15 \pm 6.09$ , respectively (Figure 7). When the test conditions changed to 600 N–40 Hz–360 min, the wear volumes ( $10^{-4} \text{ mm}^3$ ) for  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite grease were  $18.86 \pm 2.14$ ,  $77.40 \pm 5.03$ , and  $41.26 \pm 6.21$ , respectively. Obviously,  $\alpha$ -ZrP grease worked steadily and had the lowest wear volume among the three additives after a long-duration experiment. Graphite grease had better anti-wear performance than that of MoS<sub>2</sub> grease. The average friction coefficient for  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite exhibited analogous trends.



**Figure 7.** The wear volume and mean friction coefficient for the grease with  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite under long duration test (SRV test, load 600 N, temperature 80 °C, duration 360 min).

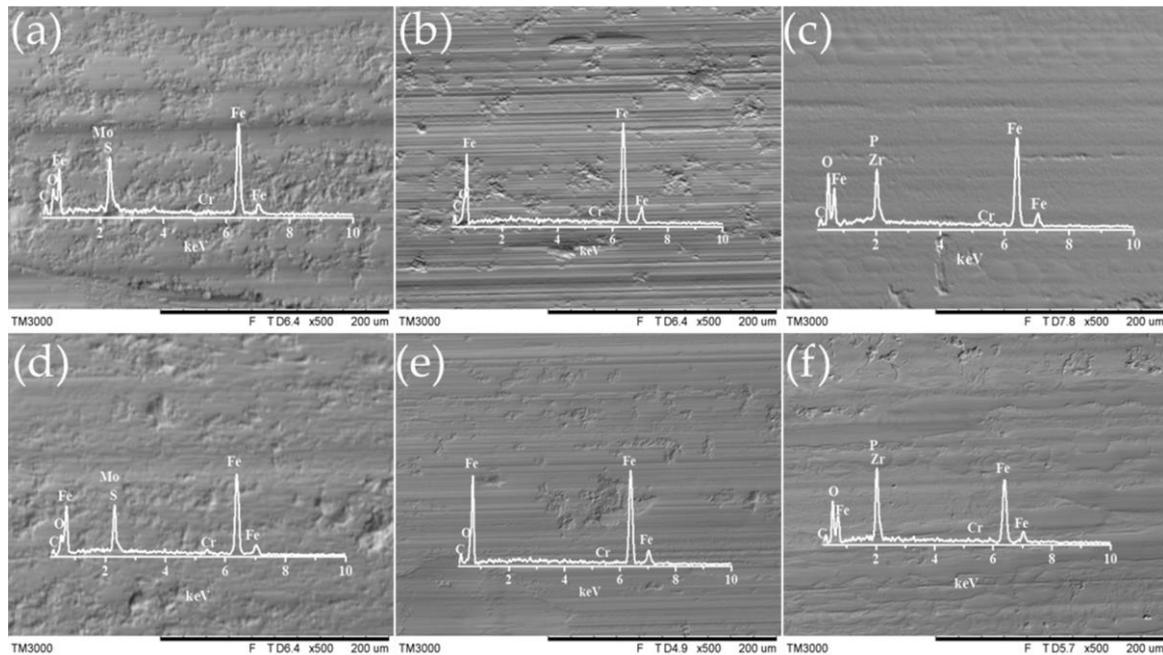
To better understand the tribological behavior of the long duration experiments, Figure 8 presents the 3D surface profiles of the corresponding wear tracks on discs after 360 min friction tests. It is obvious that the wear tracks lubricated by  $\alpha$ -ZrP grease are small and narrow at the running frequency of 20 Hz or 40 Hz. The wear tracks with MoS<sub>2</sub> and graphite grease are relatively wider and deeper.



**Figure 8.** 3D microscopic images of wear tracks on the lower disks: (a–c)  $\alpha$ -ZrP, MoS<sub>2</sub>, graphite at 20 Hz; (d–f)  $\alpha$ -ZrP, MoS<sub>2</sub>, graphite at 40 Hz (SRV test, load 600 N, temperature 80 °C, duration 360 min).

SEM micrographs and EDS spectra of the lower disks are shown in Figure 9. It can be found that the worn surface lubricated by  $\alpha$ -ZrP grease was smoothest and no sharp furrows were present on the wear track, which demonstrates that  $\alpha$ -ZrP shows the best friction-reducing and antiwear properties, corresponding to the results of tribological test. Meanwhile, the worn surface lubricated with MoS<sub>2</sub> and graphite grease displayed grooves and scuffings. The furrows of graphite grease were shallower than that of MoS<sub>2</sub>, revealing that graphite had the better antiwear properties, as discussed earlier.

From the data of the EDS spectra, the feature elements (Zr, P) of  $\alpha$ -ZrP were discovered on the wear surface. Additionally, the phenomenon was also found on the disc surface of MoS<sub>2</sub> grease. Although the EDS results showed the appearance of  $\alpha$ -ZrP and MoS<sub>2</sub> particles on the rubbing surface,  $\alpha$ -ZrP can be more effective to alleviate abrasive wear than MoS<sub>2</sub>.



**Figure 9.** Scanning Electron Microscopy (SEM) images and Corresponding Energy Dispersive X-ray Spectroscopy (EDS) spectra of the worn surfaces on the lower disks: (a–c)  $\alpha$ -ZrP, MoS<sub>2</sub>, graphite at 20 Hz; (d–f)  $\alpha$ -ZrP, MoS<sub>2</sub>, graphite at 40 Hz (SRV test, load 600 N, temperature 80 °C, duration 360 min).

#### 4. Conclusions

In this study, the tribological properties of anhydrous calcium grease with  $\alpha$ -ZrP, MoS<sub>2</sub>, and graphite particles have been researched via reciprocating and rotational motion. Based on the results obtained above, the following conclusions are listed:

1.  $\alpha$ -ZrP is considered to be a promising candidate for lubricant additives because of its outstanding lubricating properties. The maximum applied load and operating frequency for  $\alpha$ -ZrP grease can reach up to 900 N, 70 Hz, respectively.  $\alpha$ -ZrP grease can run steadily throughout the test and hold the lowest wear volume regardless of severe contact condition.
2. SEM-EDS and 3D analyses indicate that the outstanding performance of  $\alpha$ -ZrP grease comes from a stable and compact tribofilm with low shearing strength, which seems stronger and more durable than MoS<sub>2</sub> and graphite.

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**Author Contributions:** Jinxiang Dong, Hong Xu and Yingjing Dai conceived and designed the experiments; Yingjing Dai performed the experiments and analyzed the data; Wenxing Niu and Xiaosheng Zhang contributed materials; Yingjing Dai and Hong Xu wrote the paper.

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

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