



## Article

# Tribological Properties of Al<sub>2</sub>O<sub>3</sub> Nanoparticles as Lithium Grease Additives

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**Abstract:** The tribological properties of Lithium grease specimens with different concentrations of Al<sub>2</sub>O<sub>3</sub> nanoparticles were investigated using a pin on disc apparatus under different sliding speeds and normal loads. Results showed that Al<sub>2</sub>O<sub>3</sub> nanoparticles enhanced the tribological properties of lithium grease and reduced the COF and wear scar width by approximately 57.9% and 47.5% respectively.

**Keywords:** frictional coefficient; Al<sub>2</sub>O<sub>3</sub> nano additives; lithium grease lubricant



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## 1. Introduction

Lubricating greases are employed in many types of machinery. Therefore, to improve the efficiency and performance of machines and enhance the quality of products, the development and introduction of high-efficiency lubricants are required. A major advantage of using nanoparticles in greases is that they enhance lubricity. Nano-based grease provides some technical benefits in extreme conditions of high temperature and pressure because of the high surface-area-to-volume ratio of nanoparticles, which provides improved thermal conductivity, heat transfer coefficient, high thermal stability, high mobility and effective interaction between contacting surfaces.

In recent years, the use of nanomaterials has emerged as a solution to enhance the tribological characteristics of commercial lubricants/greases to ensure the efficient operation of machines. Preliminary studies have suggested improved performance in extreme pressure settings, as well as antiwear and friction reducing properties of such greases [1,2]. In lubricating oils, the use of fine metal powders (Fe, Ni, and Cu) [3], Al<sub>2</sub>O<sub>3</sub> nanoparticles [4,5], SiO<sub>2</sub> nanoparticles [6,7], and ZrO<sub>2</sub>/SiO<sub>2</sub> nanocomposites [8,9] has been shown to be effective. A lubricating grease containing nanoparticles possesses many advantages, as it acts on the surface of the friction pair, has a large specific surface area, high diffusion, easy sintering, and offers melting point reduction. One recent study [10] reported reductions of COF and wear rate of up to 20% and 42%, respectively, when SiO<sub>2</sub> nanoparticles were dispersed in paraffin grease.

The influence of adding nanoparticles to lithium grease on the friction coefficient was investigated in earlier studies [11,12]. Nano titanium, silicon and hybrid nano oxides at five concentration levels were used to disperse the lithium grease [13] and it was observed that the friction coefficient decreased by 50% when 6% wt. hybrid nanoparticles of TiO<sub>2</sub>/SiO<sub>2</sub> were applied, compared to either 6 wt.% TiO<sub>2</sub> (27%) or 8 wt.% SiO<sub>2</sub> (46%) alone. The tribological properties of lithium grease with graphene layer additives were also investigated in [14]. That study showed that the reduced friction coefficient and wear resulting from the use of lithium grease was mainly attributed to its large specific surface area, larger number of active groups, and typical layered structure. The tribological

and rheological behavior of Al<sub>2</sub>O<sub>3</sub> nanorods as an additive in lithium grease at different concentrations was investigated in [15]. The correlation between the rheological properties and the antiwear and friction-reducing mechanism of nanorod–Al<sub>2</sub>O<sub>3</sub> grease was also evaluated in this study. The antifriction and surface reconditioning properties of Al<sub>2</sub>O<sub>3</sub> nano-additive lithium grease were investigated in [16]. A MRH-3 friction and wear tester was used to investigate the tribological properties of various types of nanoparticles as lithium grease additives; the results suggested that the addition of Al<sub>2</sub>O<sub>3</sub> nanoparticles improved the antifriction properties of the base grease surface reconditioning ability [17].

The vibration and friction coefficients of modified Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> composite nanoparticles were also examined through four-ball and thrust-ring tests [18,19]. The tribological investigation of oils with nano-additives showed that the application of Fe, Cu, and Co nanoparticles decreased the coefficient of friction and wear between friction pairs [20]. Carbon nanotubes (CNTs) have also been used as nano additives to lithium grease to study the vibration behavior of ball bearings. The vibration amplitude was improved with increasing concentrations of CNTs [21]. The tribological properties of lithium grease dispersed with CNTs were evaluated with a four-ball tester. The results showed around 63% and 81.5% reductions in the wear scar and friction coefficients, respectively [22].

The antiwear properties of nanoparticles suspended in a polyalphaolefin (PAO<sub>6</sub>) were studied [23,24], applying CuO, ZnO and ZrO<sub>2</sub> nanoparticles dispersed at 0.5 wt.%, 1.0 wt.% and 2.0 wt.% in (PAO<sub>6</sub>). The copper nano powder additive altered the worn surface topology and did not impair the lubrication characteristics of the SAE-30 motor oil [25]. The wear and friction properties of surface-modified Cu nanoparticles as 50 CC oil additives were studied [26]. A disc-on-disc type tester was used to examine the role of fullerene nanoparticles dispersed in a mineral oil-based lubricant [27].

In this study, the tribological properties of lithium grease consisting of different concentrations of Al<sub>2</sub>O<sub>3</sub> nano-additives were examined under diverse operating conditions. The correlation between the coefficient of friction and the wear of the Al<sub>2</sub>O<sub>3</sub> lithium grease specimens was evaluated in this study. The COF of Al<sub>2</sub>O<sub>3</sub> lithium grease specimens were compared to those described in other studies using TiO<sub>2</sub>, SiO<sub>2</sub> and hybrid TiO<sub>2</sub>/SiO<sub>2</sub> nanoparticles, and in similar studies using Al<sub>2</sub>O<sub>3</sub> nanoparticles. In addition, variations in the contact temperature of the specimen with different contents of Al<sub>2</sub>O<sub>3</sub> nanoparticles and sliding times were evaluated. This current research can be used as a reference for the use of nanoparticles as grease additives for the reduction of friction. The goal is to identify the optimal concentration of Al<sub>2</sub>O<sub>3</sub> nano-additives in order to maximally reduce COF and wear scar.

## 2. Experimental

### 2.1. Material

Aluminum oxide nanoparticles (Al<sub>2</sub>O<sub>3</sub>, 80% alpha, 20% gamma, purity 99.9%) with an average diameter of 50 nm and specific surface area 35 m<sup>2</sup>/g were purchased from US Research Nanoparticles, Inc., and lithium grease was purchased from a local company in Egypt. Figure 1 shows a SEM image of the morphology of the purchased Al<sub>2</sub>O<sub>3</sub> nanostructures.

Figure 2 shows the XRD pattern of Al<sub>2</sub>O<sub>3</sub> nanoparticles, obtained using a Bruker D8 X-ray diffractometer (Bruker, Billerica, MA, USA). The radial scan intensity versus scattering angle (2θ) recorded at room temperature with anode material copper and filament current 30 mA is shown. To calculate the d-value, Bragg's law was applied.

$$n\lambda = 2D \sin\theta \quad (1)$$

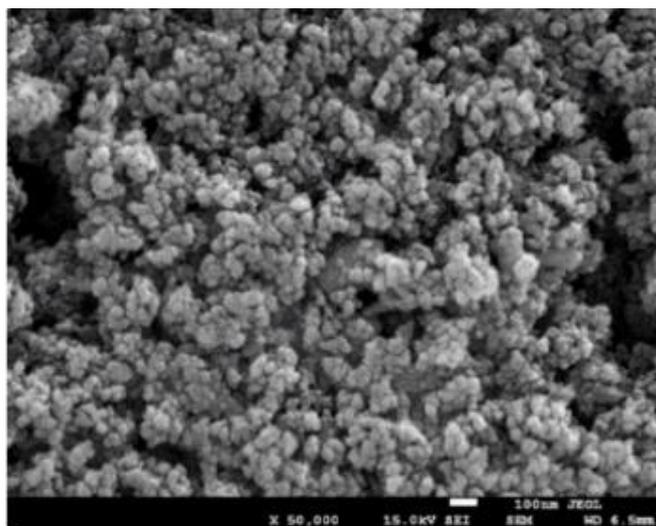


Figure 1. SEM image of Al<sub>2</sub>O<sub>3</sub> nanoparticles.

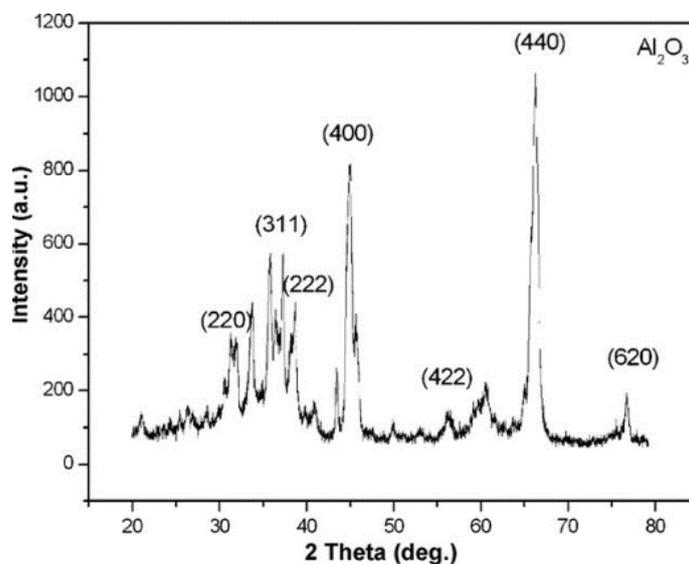


Figure 2. XRD pattern of Al<sub>2</sub>O<sub>3</sub> nanoparticles.

## 2.2. Samples Preparation

Samples were prepared using pure lithium grease and Al<sub>2</sub>O<sub>3</sub> nanoparticles (50 nm) at concentrations of 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.%, and 10 wt.%. In this study, lithium grease was chosen because it is one of the most common and cheapest greases, and is ideal for metal to metal applications. Additionally, lithium grease is stable at a range of temperatures, which makes it ideal for a variety of applications, such as in automotive and household products.

The pretreatment of Al<sub>2</sub>O<sub>3</sub> was performed as follows: Al<sub>2</sub>O<sub>3</sub> nanoparticles were sonicated by immersion in 10 wt.% sodium hydroxide solution with magnetic stirring for 1 h, and then in acetone with stirring for 1 h, with the aim of cleaning their surface to eliminate any impurities. After that, the powders were filtered and washed with distilled water and dried in an electrical furnace at 110 °C for 1 h. An ultrasound sonicator, Q700 High Volume Continuous Floccell (Qsonica, Newtown, CT, USA) was used to disperse and de-agglomerate the nanoparticles in the lithium grease. The Q700 Sonicator was utilized because of its accuracy, simplicity, and yield. The optimal sonication process parameters, such as frequency, amplitude, probe type and depth, and duration were identified and applied.

### 2.3. Tribology Test Rig

A MM-W1A Friction and Wear Testing Machine (Jinan Chenda Testing Machine Manufacturing, Jinan, Shandong, China) was used to examine the tribological properties of all samples, meeting the SH/T 0189-1992 Lubricant Anti-wear performance evaluation method (Four-ball Tester Method) requirements and conforming to the ASTM D4172—94 and ASTM D 5183—95 standards. An image of the MM-W1A and the equipment used to collect the data is shown in Figure 3.



**Figure 3.** MM-W1A Friction and Wear Testing Machine.

During the tests, a stainless-steel ball was pushed on a rotating aluminum disc to apply a load, as illustrated in Figure 4. All tests were conducted at room temperature, and 1 gm Li-based grease was used in each test. The applied force varied from 25 to 100 N, and the sliding speeds from 250 to 1000 rpm. The surface roughness of the aluminum disc was measured to be ( $R_a = 0.072 \mu\text{m}$ ,  $R_q = 0.094 \mu\text{m}$ , and  $R_z = 0.578 \mu\text{m}$ ). The coefficient of friction ( $\mu$ ) for the pin-disk friction pair was then calculated as follows:

$$F = \mu p / 3 \quad (2)$$

$$T = 3 \times Fr \quad (3)$$

$$\mu = T / p r = 0.043332 T / p \quad (4)$$

$F$  = Friction force [N]

$P$  = Axial force [N]

$T$  = Friction moment [N.mm]

$r$  = contact point trace radius of big ring and pin =  $46.155/2$  [mm]

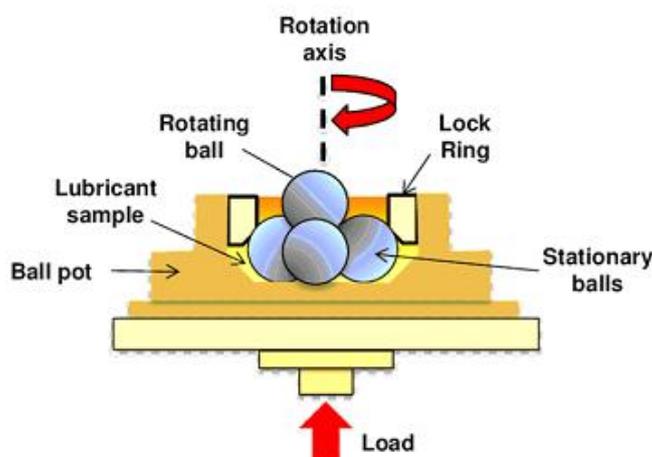


Figure 4. Schematic view of the friction pair assembly.

### 3. Results and Discussion

#### 3.1. Influence of $\text{Al}_2\text{O}_3$ Nanoparticles on the Friction Coefficient

The COF and subsequent wear scar depths of the samples were measured and are discussed in this section. The instantaneous variation of COF for a sample containing 2.0 wt.% of  $\text{Al}_2\text{O}_3$  nanoparticles with changing loads and a constant speed of 200 rpm is shown in Figure 5A. Each test was repeated three times for accuracy, and the COF was measured every 20 min to obtain the instantaneous data which was then fitted (Figure 5B) to better visualize the trends. The results showed that the addition of  $\text{Al}_2\text{O}_3$  nanoparticles improved the antifriction properties of the base grease surface. The fluctuation in the COF was due to the accumulation of wear debris and the agglomeration of  $\text{Al}_2\text{O}_3$  nanoparticles. The friction coefficient data were fitted using the polynomial least square method, which was suitable for such a friction coefficient trend.

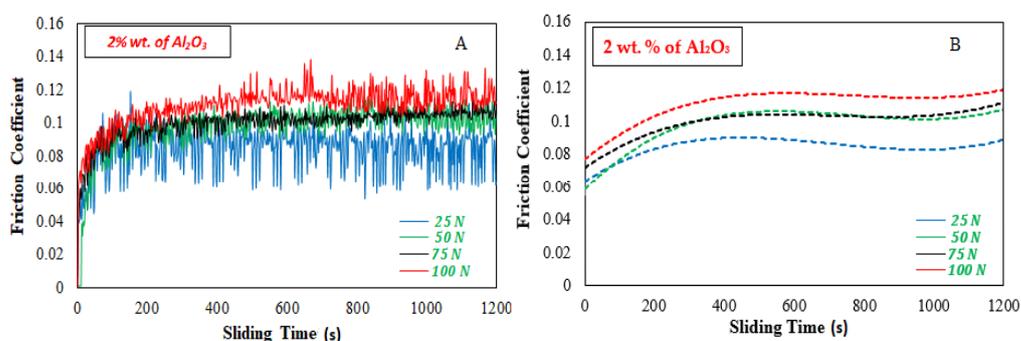
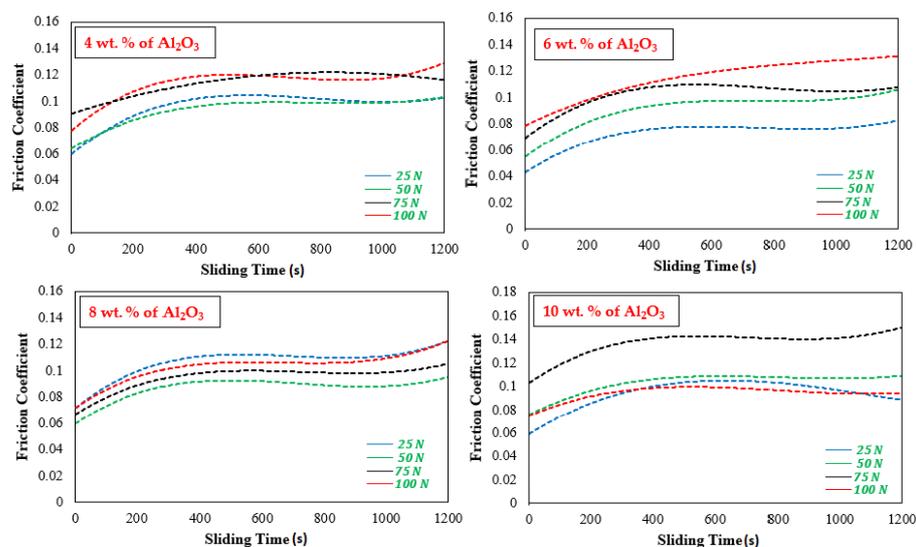


Figure 5. Variation of COF of 2 wt.%  $\text{Al}_2\text{O}_3$  nanoparticle-modified specimens at a constant speed of 200 rpm. (A) instantaneous data; (B) data fitted.

Using the same method, the COF of samples containing 4 wt.%, 6 wt.%, 8 wt.% and 10 wt.%  $\text{Al}_2\text{O}_3$  nanoparticles were measured and similar trends were observed. A summary of all results at varying load and 200 rpm is shown in Figure 6. The results show that the COF of  $\text{Al}_2\text{O}_3$  modified nanoparticles specimens were lower than that of the base grease specimen, and that the specimen containing 4 wt.% was the most effective. This implies that  $\text{Al}_2\text{O}_3$  as a nano-additive can significantly reduce the interfacial friction and improve the bearing capacity of mechanically moving part. Therefore, grease containing  $\text{Al}_2\text{O}_3$  nano-additive has great potential.



**Figure 6.** COF of Li/ $\text{Al}_2\text{O}_3$  specimens for constant speed of 200 rpm with data fitted at 4 wt.%, 6 wt.%, 8 wt.%, and 10 wt.%.

The COF of samples containing 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.% and 10 wt.%  $\text{Al}_2\text{O}_3$  nanoparticles at varying load and speed conditions are shown in Figure 7. Figure 7A shows the variations of COF for all samples with changing load and a constant speed of 1000 rpm. It was observed that the COFs of all modified grease specimens were less than that of the base grease specimen. A reduction of 57.9% in COF, compared to the pure grease specimen, was observed with 4.0 wt.%. Higher concentrations of  $\text{Al}_2\text{O}_3$  nanoparticles resulted in lower COFs because, they cause the contact surface to become rough, leading to decreased lubrication. Hence, the optimum amount of  $\text{Al}_2\text{O}_3$  nanoparticles was found to be 4.0 wt.%.

The variation of COF of  $\text{Al}_2\text{O}_3$  nanoparticle-modified specimens at a constant load of 50 N and with varying speeds is presented in Figure 7B, which shows that COF decreases as rotational speed increases. In all results presented in Figure 7A,B, it should be noted that the COFs of all modified samples were significantly lower than that of the base grease specimen, which confirms the potential of  $\text{Al}_2\text{O}_3$  nanoparticles to minimize COF. This is mainly attributed to the rolling effect of nanoparticles between the rubbing surfaces, which results in a decrease in the contact pressure, as illustrated in Figure 8. In addition, having  $\text{Al}_2\text{O}_3$  nanoparticles in the specimens introduced an excellent mending effect and helped repair worn surfaces due to its high specific surface area and small size [28–31]. The formation of thin layer between asperity contact surfaces is also an important factor, as it prevents metal-to-metal contact [32–36].

The MM-W1A testing machine contained a heating system that enabled instantaneous contact temperature measurements for the friction pair using platinum thermo-resistance. Figure 9 shows the variations of the contact temperature above the ambient temperature of the specimen versus sliding time with different contents of  $\text{Al}_2\text{O}_3$  nanoparticles for a test time of 20 min at 50 N constant load and 1000 rpm constant speed. It can be seen that the frictional surface contact temperature of the  $\text{Al}_2\text{O}_3$  nano-modified specimens was lower than that of the base grease specimen, due to the thermal conductivity characteristic of the nano  $\text{Al}_2\text{O}_3$ . Figure 9 also shows that the contact temperature of the 4 wt.%  $\text{Al}_2\text{O}_3$  nanoparticles specimens and base grease specimen exhibited similar behavior for the entire sliding time, i.e., about 9 °C reduction in contact temperature, which indicates that the lithium grease specimen was only stable when 4 wt.%  $\text{Al}_2\text{O}_3$  nano-modified was added. Specimens with concentrations other than 4 wt.% showed different behavior, compared to base grease specimen.

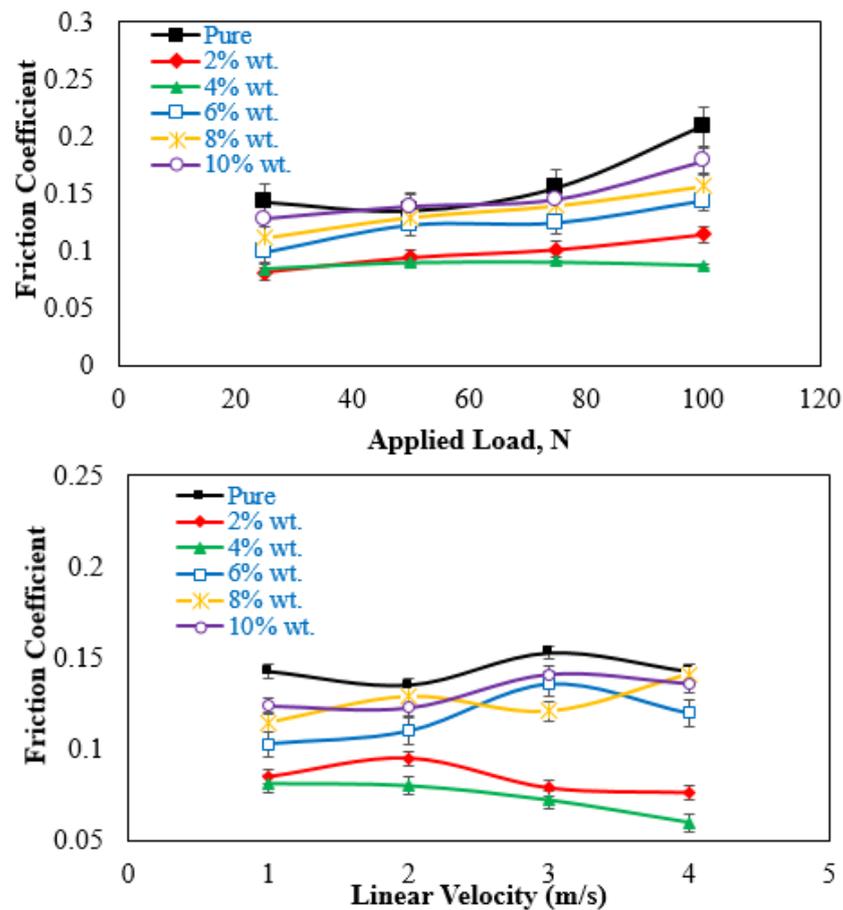


Figure 7. Average friction coefficients of Al<sub>2</sub>O<sub>3</sub> nanoparticles samples at different loads and speeds.

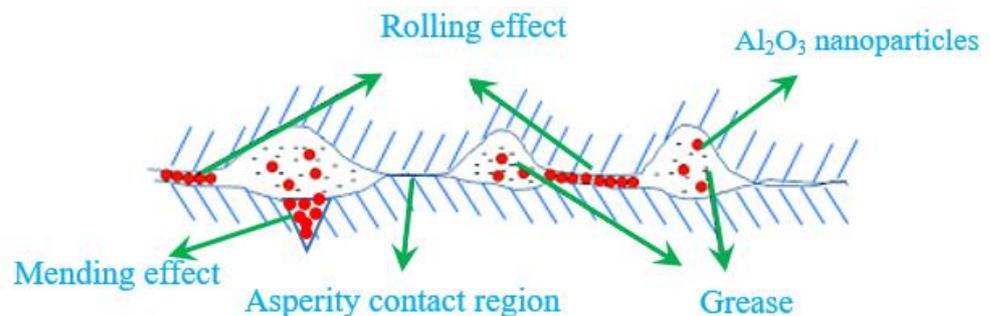
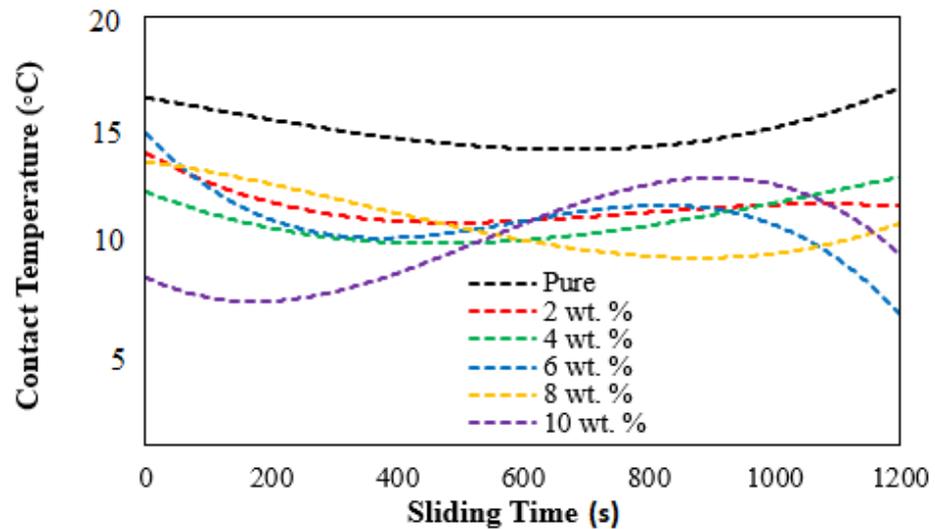


Figure 8. Schematic of rubbing surfaces mechanism in the grease with dispersed nanoparticles.

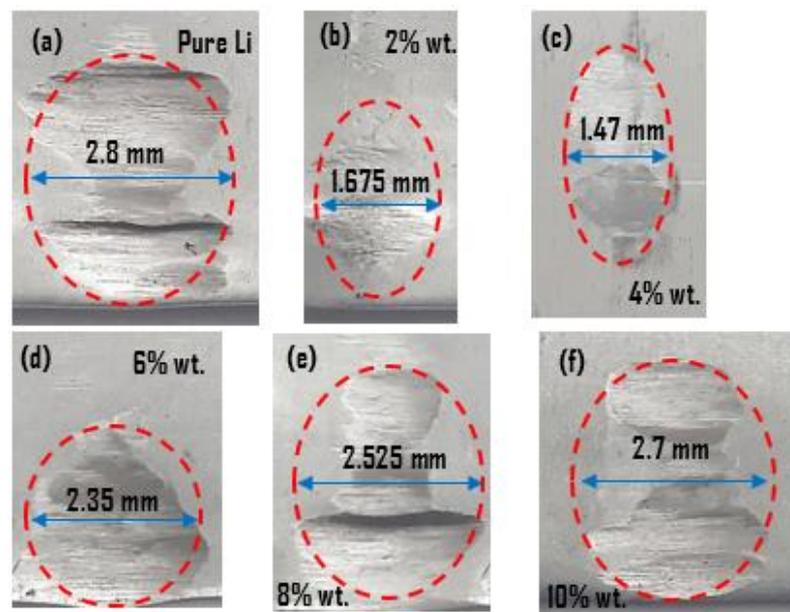
A comparison was performed of the performance (at varying loads and speeds) of our lubricant containing 4 wt.% Al<sub>2</sub>O<sub>3</sub> nanoparticles with other nano-additives reported in literature [13]. It was observed that the use of 4 wt.% Al<sub>2</sub>O<sub>3</sub> nanoparticles resulted in a significant reduction in COF as compared to other additives (SiO<sub>2</sub>, TiO<sub>2</sub> and hybrid SiO<sub>2</sub>/TiO<sub>2</sub> nanoparticles) despite being used in larger quantities. In [13], the highest reduction in COF was reported to be approximately 34%, using SiO<sub>2</sub> nanoparticles. In contrast, in current study, the grease specimen with only 4 wt.% Al<sub>2</sub>O<sub>3</sub> nanoparticles decreased the COF by 42.9% at low speeds and 57.9% at high speeds, thereby confirming the viability and effectiveness of this method. Adding Al<sub>2</sub>O<sub>3</sub> to lithium grease remarkably improved its tribological properties, and increased the load bearing capacity. The enhanced performance of the lithium grease was due (among others) to the increased surface area to volume ratio and repairing lubricated surfaces by filling in wear tracks.



**Figure 9.** The variation of the contact temperature for the grease at different contents of  $\text{Al}_2\text{O}_3$  nanoparticles with test time.

### 3.2. Influence of $\text{Al}_2\text{O}_3$ nanoparticles on Wear Scar

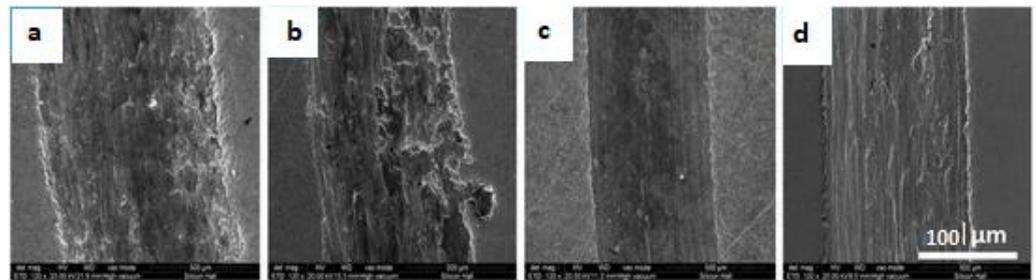
To observe the effect of friction, we measured the wear scar width using an optical microscope. Figure 10 shows the wear scar images of lithium grease with and without  $\text{Al}_2\text{O}_3$  nanoparticles at an applied load of 100 N and a constant speed of 200 rpm. It was observed that the wear scar width of the specimen using 4 wt.%  $\text{Al}_2\text{O}_3$  nanoparticles was about 1.47 mm, in contrast to the 2.8 mm of the pure lithium grease specimen and representing a reduction of around 47.5%.



**Figure 10.** Wear scar images on aluminum plate of  $\text{Al}_2\text{O}_3$  nanoparticle-modified specimens. (a) Pure Li, (b) 2 wt.%, (c) 4 wt.%, (d) 6 wt.%, (e) 8 wt.%, (f) 10 wt.%.

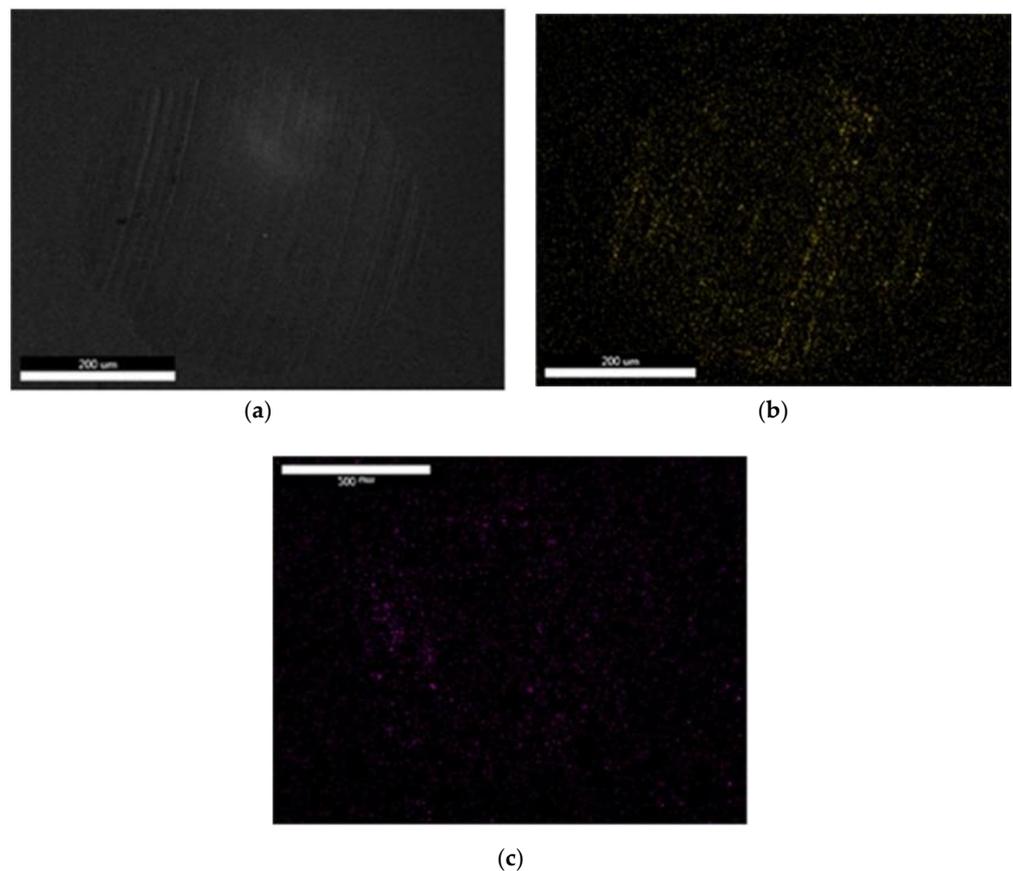
Four SEM disk specimen wear track images are shown in Figure 11. Wear tracks for the pure lithium, 2 wt.%, 4 wt.% and 6 wt.% were investigated. Worn material debris was investigated on the wear track for the four samples. The pure lithium (a), 2 wt.%  $\text{Al}_2\text{O}_3$  (b), and 6 wt.% specimens suffered severe wear. The specimen with 4 wt.% (c) showed a

smooth wear track which did not have any deep scratches, significant plastic deformation, or deep sliding direction marks.



**Figure 11.** SEM specimen wear tracks images at 100  $\mu\text{m}$ . (a) pure Li, (b) 2 wt.%, (c) 4 wt.%, (d) 6 wt.%.

An energy dispersive spectrometer (EDS) was used to assess the overlay of the worn steel surfaces. Figure 12 shows an EDS image of element overlay of the worn steel surface lubricated with lithium grease containing 4 wt.%  $\text{Al}_2\text{O}_3$  nanoparticles.

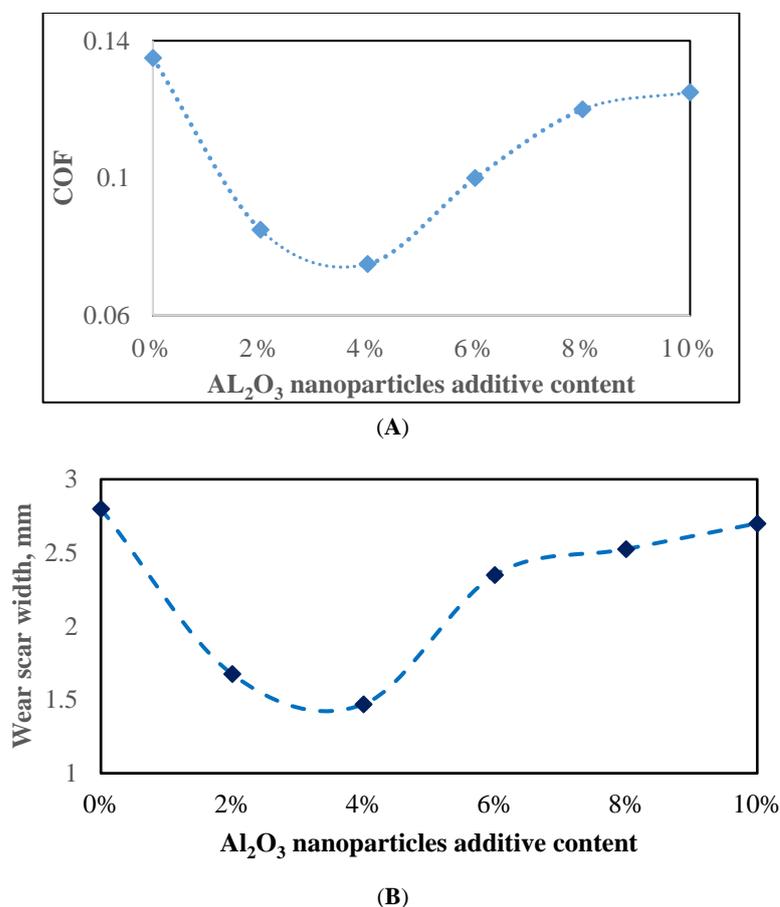


**Figure 12.** EDS images of the worn steel surface for (a) initial state; (b) enrichment of Al and (c) enrichment of O.

It can be seen that when the  $\text{Al}_2\text{O}_3$  was added to the lithium grease, the overlay of Al and O elements on the worn steel surface was detected, implying that the  $\text{Al}_2\text{O}_3$  had been incorporated into the surface layer through adsorption or deposition, leading to reduced friction and wear of the tribo-pairs.

Figure 13A shows the behavior of the friction coefficient with the increase of  $\text{Al}_2\text{O}_3$  content. The friction coefficient gradually decreased, but when the content reached a

certain value, it started to rise again. Figure 13B shows that the behavior of the wear scar diameter was similar to that of the friction coefficient, confirming the interrelation between such variables.  $\text{Al}_2\text{O}_3$  nanoparticles can be considered as effective antiwear additives due to the formation of thin-films on the worn surfaces to prevent metal-to-metal contact. It is believed that the observed increment in the wear scar diameter and the friction coefficient for contents above 4 wt.% was caused by the agglomeration of the additives.



**Figure 13.** Friction coefficient and wear scar diameter and versus  $\text{Al}_2\text{O}_3$  nanoparticle additive content. (A) COF, (B) Wear scars width.

#### 4. Conclusions

Lithium grease containing different concentrations of  $\text{Al}_2\text{O}_3$  nanoparticle was tested for COF at different loads and sliding speeds. It was found that the grease specimen with 4 wt.% of  $\text{Al}_2\text{O}_3$  reduced the COF by up to 57.9% compared to the base grease specimen, i.e., much more than the reduction reported in other studies using  $\text{TiO}_2$ ,  $\text{SiO}_2$  and hybrid  $\text{TiO}_2/\text{SiO}_2$  nanoparticles [13] and in similar studies using  $\text{Al}_2\text{O}_3$  nanoparticles [15]. Considerable reduction in COF and up to 47.5% reduction in wear scar confirms the potential of using  $\text{Al}_2\text{O}_3$  nanoparticle in lithium-based greases.

**Author Contributions:** Conceptualization, A.N. and J.A.; methodology, A.R. and N.M.G.; software, A.N.; validation, A.R., N.M.G. and M.D.H.; formal analysis, N.M.G.; investigation, A.N.; resources, A.R.; data curation, A.N.; writing—original draft preparation, N.M.G.; writing—review and editing, J.A.; visualization, M.D.H.; supervision, J.A.; project administration, N.M.G. All authors have read and agreed to the published version of the manuscript.

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