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Research Article

Excellent current-carrying tribological performance of liquid-like carbon dots in lubricating grease

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Abstract

When exposed to the electric current environment, the motor bearings in electric equipment often endure severe wear or arc erosion. Hence, liquid-like carbon dots (LCDs), an advanced conductive additive with great dispersibility and low-addition effectiveness, was designed and introduced into lithium-based grease to address the aforementioned current-carrying problem. LCDs could form a conductive network in grease to provide an effective electric current transport pathway, which improves the current-carrying tribological properties. Additionally, the ionic structure enables LCDs to easily adsorb on the substrate under current-carrying conditions and form a protective film. Thereby, this work not only provides some new insights into the current-carrying lubrication mechanism of conductive additives for grease, but also expands the potential application of LCDs in electric machinery.

Keywords: Conductive lubricating grease; Lithium-based grease; Liquid-like carbon dots; Conductive network; Current-carrying friction.

1. Introduction

Due to the increasing demand for low-emission and energy-efficient commuting, the electric equipment industry is considered a significant and potential field [1-3]. The electric motor bearing is a crucial component of electric equipment, transmitting rotational force and ensuring the stable operation of the electric motor. However, under the electrical environment, some issues, such as bearing lubrication failure and electrical erosion, could be caused by the current-carrying friction [4-6]. Generally, there are two methods to prevent the current-carrying friction: bearing insulation and bearing conductivity. For the former, high-quality insulating coatings or insulated ceramic bearings can block the current conduction. But the cost of these methods is relatively high, making them unsuitable for large-scale production. For the latter, conductive lubrication grease is one of the commonly used methods, conductive grease has been the subject of extensive research as a potential method to solve the problem of current-carrying friction, due to its minor optimization adjustments, low production costs, and effectiveness in current-carrying friction issues [4, 7-9]. Conductive grease is obtained by incorporating the conductive filler, such as metal powder, carbon-based materials, ionic liquids, conductive polymers, etc.[10, 11] into the base grease [12-14]. These conductive fillers form a conductive network in the grease to conduct current and prevent the accumulation of charges. Reducing the damage caused by thermal effects, such as Joule heat, while maintaining excellent anti-friction and anti-wear performance [15].

In carbon-based materials, graphite [16], graphene [17], conductive carbon black(CB) [18, 19] and carbon nanotubes [20, 21], possess good electrical conductivity, which is widely studied in the field of current-carrying lubrication. For example, the electrical conductivities and tribological properties of conductive grease prepared by graphite or CB are improved [19, 22]. Moreover, the graphene nanoparticles with varying specific surface areas as additives in poly alpha olefin (PAO) improve the current-carrying friction properties of PAO [17]. The carbon nanotubes (CNTs) as additives in lubricating grease exhibit great electrical conductivity and excellent current-carrying tribological performance [12]. Although carbon-based nanomaterials are considered ideal conductive lubricants due to their excellent electrical conductivity and lubricating properties, the poor dispersibility in grease and high additive concentrations limit their application [18].

Carbon dots (CDs), as a novel zero-dimensional carbon nanomaterial, have gained more attention because of the cost-effectiveness, good conductivity and superior lubricating properties [23, 24]. For instance, as additives in PAO [25, 26], PEG [27], water [28, 29] or lubricating grease [30], CDs can effectively enhance the tribological performance with lower concentration. Simultaneously, CDs as additives exhibit electrically responsive tribological properties, demonstrating the potential electrical conductivity

For example, using chitosan polyamines as raw materials, CDs modified with polyelectrolytes were synthesized, which can achieve friction regulation under the action of an external electric field as low as ± 1.0V[31]. In addition, Poly-ionic liquid functionalized carbon dots (CDS-PIL) were synthesized using polyethyleneimine-grafted carbon nanotubes, which exhibited excellent potential-controlled friction performance in PEG 200[32]. As solid additive, the dispersibility of CDs as additives in conductive grease is better than other carbon-based materials, but it still needs to be improved.

In particular, liquid-like carbon dots (LCDs), as a special type of CDs, possess the fluidity at room temperature and excellent tribological properties. For instance, the bilayer carbon nanofluid prepared from chitosan can achieve intelligent regulation and lubrication under the action of an external electric field [33]. In addition, solvent-free carbon dots-based ionic fluids were prepared using citric acid and poly(sodium 4-styrene sulfonate) as raw materials. These fluids have good dispersibility in PEG and can effectively improve the tribological properties of PEG[34]. Through combining adenine (ADS) with low-concentration PDDA and using a rapid, low-energy microwave method, PDDA modified carbon dot-based solvent-free nanofluid was prepared. When used as water-based additive, it significantly reduces wear and heat generation[35]. In our previous research, LCDs synthesized by pyrolysis method not only show good dispersion performance in lubricating grease but also improve the lubricating properties[36, 37].

Hence, in this work, to improve the dispersibility in base grease, the LCDs with fluidity and nano-structure were obtained by pyrolysis method [36]. The current-carrying tribological performance and mechanisms of LCDs in lithium-based grease were investigated under the condition of electrification. Meanwhile, an electro-rheometer and a volume resistivity meter were employed to test the electrorheological adaptability and electrical conductivity of the grease samples. Combined with the SEM results of the soap fiber structure, it was verified that LCDs can be uniformly dispersed within the grease and form a conductive network. In addition, the influences of additive concentration and temperature fluctuations on the lubricating properties were also studied. This study not only expands the application horizons of CDs, but also provides valuable insights for using CDs as conductive lubricant additives in both research and practical applications.

2. Experimental details

2.1. Materials

Citric acid was purchased from Aopusheng Chemical Ltd (Tianjin, China). Tetrabutylammonium hydroxide (H_2O content of 25 wt%) was obtained from the Aladdin Reagent Co., Ltd (Shanghai, China). PAO6 was bought by Sinopec (Maoming, China). Lithium 12-dodecyl stearate was obtained from Hongxing Chemical Co., Ltd (Shandong, China). Phosphoric acid was provided by Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Deionized water was used throughout the experiment, which was prepared by an ultrapure water purifier in our laboratory. All chemicals were used as received without further purification.

2.2 Preparation

Synthesis of $[N_{4444}][H_2PO_4]$: Prepared $[N_{4444}][H_2PO_4]$ by acid-based neutralization reaction. Firstly, weight 9.8 g phosphoric acid(1 mol) into a flask and dilute with a small amount of water. Then dorp 1 mol tetrabutylammonium hydroxide under magnetic stirring, and react under closed conditions for 12 hours. Finally, the final product is obtained by rotary evaporation and drying.

Synthesis of LCDs: First, 10 g Citric acid(CA) and 2 g $[N_{4444}][H_2PO_4]$ were weighed in a beaker and reacted at 200 ℃ until the liquid turned reddish-brown. Then, 80 ml of deionized water was added and allowed to stand for 3 hours, followed by an ultrasound for 10 minutes to promote the dissolution of unreacted CA. Subsequently, 80 ml of ethyl acetate was added for extraction and separation. Finally, LCDs were obtained by spin evaporation and drying[36]. The process of synthesizing LCDs is shown in **Fig. 1a**. The mass spectrometry (MS) results of LCDs are shown in **Fig. S1**, it can be observed that in the cation mass spectrum, there is a distinct peak at around 242, corresponding to $[N_{4444}]$. Furthermore, in conjunction with the material analysis section of the references mentioned above, the presence of alkyl peaks and C-N^+ in the material was confirmed through FTIR and XPS. It can be concluded that the LCDs possess an ionic structure and the material was synthesis successfully.

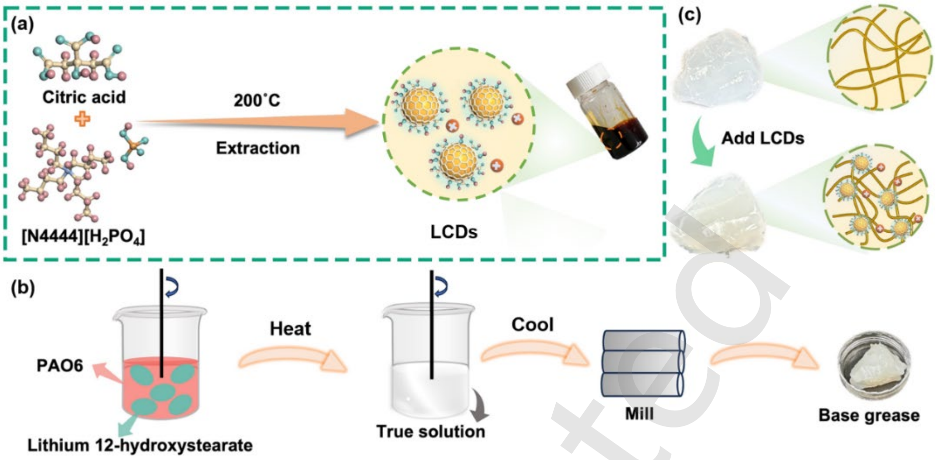

Fig. 1 The synthesis process and schematic diagram of experiment samples (a) LCDs, (b) base grease, (c) Physical state and microstructure of base grease and grease with 0.25wt% LCDs.

Synthesis of base grease: A certain ratio of lithium dodecyl alcohol was introduced into a beaker, followed by the addition of PAO6. After heating and stirring until a true solution is formed, then conducted for five minutes. The beaker was removed, pouring another PAO6 for rapid cooling, and continued stirring until it cooled to room temperature. Finally, the base grease was obtained after grinding twice by a three-roller mill. The details of the synthesis diagram are shown in **Fig. 1b**.

Synthesis of conductive grease: A certain mass of base grease and LCDs was mixed. Then, grinding twice with the three-roller mill, conductive grease was finally obtained. After adding LCDs, the state of matter and microstructure of base grease are shown in **Fig.1c**. The physical and chemical properties of grease samples are shown in **Table 1**.

Table 1 Physical and chemical properties of grease

Samples	Drop point/°C	Penetration/0.1 mm	Volume resistivity /Ω·cm
Base grease	206	262	3.4×10^{13}
Base grease with 0.25% LCDs	208	264	1.3×10^{12}

Base grease with 0.50% LCDs	206	265	1.7×10^{12}
Base grease with 1.00% LCDs	205	258	2.6×10^{12}

2.4 Tribological test

The current tribological test was conducted in four-ball friction testing machine (Jinan Shunmao, China). Test steel balls were made by CGr15 bearing steel with a diameter of 12.7 mm. The experiment was tested under 392 N, 1200 rpm, for one hour. Furthermore, a DC power was supply. The default setting of the DC power is 5 V and 1 A. During the current-carrying friction process, due to the excessive load current, the current is maintained at the default setting value (1 A), while the voltage continuously varies during friction process. The positive electrode was connected to the rotating shaft of the upper part of the friction testing machine, and the negative electrode was connected to the test oil box. The bottom of the oil box was coated with insulation coating to prevent damage to the machine when applying current. During the current-carrying friction process, the temperature change is measured by the temperature sensing device of the four-ball friction tester. The temperature sensing device is connected to the oil box, and the current-carrying friction test is carried out at room temperature (27-30 °C). The temperature will be recorded in real time during the test.

2.5 Characterization

For the electrical performance of the grease samples, since the base grease used is a lithium-based grease, which is inherently insulating, a volume resistivity meter was employed in this study to investigate the electrical performance of the grease samples. The electric volumetric resistivity of lubricating grease samples was tested by a high resistance meter (Huace, China). The resistance of the grease samples was measured at a voltage of 1000 V, waiting for one minute until the data stabilized, and then recording the data. Then, calculated the volume resistivity of the samples using Formula [15] $\rho_v = R_x \frac{A}{h}$ (where ρ_v represents the volume resistivity, A represents the effective electrode area and the value is 0.8105 cm^2, h represents the thickness of grease samples and the value is 4 cm.)

The influence of the electric field on the structure of based grease and conductive grease was investigated using an electro-rheometer (MCR 302e, Anton Paar, Switzerland). By applying an external electric field in the oscillation mode to test the breakdown voltage and viscoelasticity. The shear rate of the test breakdown voltage is 35/s, the test temperature is 25°C, and the shear strain of the viscoelastic test is logarithmically changed from 0.01%-100%, and the test angular frequency is 10 rad/s.

The depth and roughness of the worn surface were measured through 3D profiler. Confocal mode was used for 3D testing, and BF 20 was used as the test lens. Scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), X-ray photoelectron Spectroscopy (XPS), and mass spectrometry (MS) were used to analyze the wear scar of the ball after the friction test and the structure of LCDs. The morphological and elemental changes on the wear surface were observed. In addition, the test condition for SEM and EDS is an acceleration voltage of 15 kV. Using petroleum ether to clean lithium grease and conductive greases to obtain the soap powder. Then using SEM to observe the soap fiber structure.

3. Results and discussion

3.1 Electrical conductivity of grease samples

The volume resistivity test results are presented in **Table 1**. After adding LCDs, the volume resistivity is reduced by one order of magnitude compared with base grease. This means that LCDs can improve the conductivity as grease additives to prepare the conductivity lubricating grease [38]. Compared to other conductive additives, LCDs can significantly enhance the electrical conductivity of lubricating samples (**Table S1**). As the concentration of LCDs increases, the volume resistivity of grease samples increases but is still at the same order of magnitude, which might be attributed to the aggregation of LCDs in grease.

3.2 Current-carrying tribological performance

To evaluate the friction reduction and anti-wear properties of conductive grease under electric current conditions, the current-carrying friction tests were conducted. First of all, the current-carrying tribological performance of the base grease was conducted. In **Fig. 2a-b**, the friction curve of the base grease is stable without current-carrying conditions. However, under current-carrying conditions, the friction coefficient of base grease increases significantly with obvious fluctuation. In addition, the wear scar diameter increases from 0.53 mm to 0.75 mm. These illustrate that the lithium-based grease cannot provide stable and effective current-carrying lubrication effect.

Fig. 2d and **e** present the test results of base grease with LCDs. Under the same current-carrying conditions, the addition of LCDs significantly enhances the current-carrying tribological performance of the base grease. In comparison with the base grease, the friction curve is more stable and the wear scar diameter decreases. From the friction test results, the base grease with 0.25 wt% LCDs shows the optimal current-carrying tribological performance. To further verify the current-carrying friction performance of LCDs, a comparison of LCDs with other types of additives is conducted in Table S1. From the data in the table, it can be seen that this work demonstrates more excellent conductivity and current-carrying friction performance compared with other types of conductive additives.

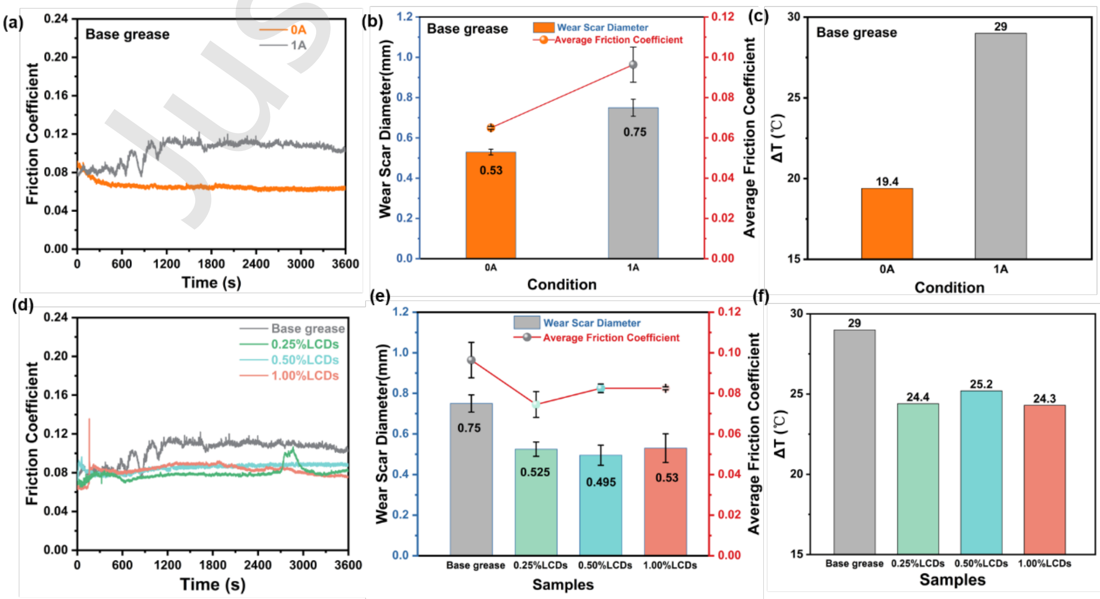

Fig. 2 (a, b, d, e) Current-carrying friction performance, (c, f) ΔT of grease samples in accordance

with a, d

Under the electric current conditions, a large amount of heat will be generated in the friction area, affecting the lubricating performance. To investigate the temperature variation (ΔT) of the current-carrying friction test, the temperature values before and after friction tests were recorded, the result was shown in Fig. 2c and Fig.2f, where the temperature rise data were obtained during the friction tests in Figures 2a and 2d. It can be seen that the ΔT of base grease increases to 29℃ when applying current, which is higher than that without current. After adding LCDs, the ΔT exhibits an obvious reduction about 5℃, indicating that LCDs reduce the system temperature under the electric current conditions. The reason for this phenomenon is that LCDs can provide conductive pathways to reduce the heat generation caused by current.

3.3 Electrorheological performance

To explore the effect of the electric field on the electrorheological properties and viscoelastic performance of base grease and base grease containing LCDs, the electrorheological tests were studied, as shown in **Fig. 3**. The breakdown voltage test results are displayed in **Fig. 3a**, In the breakdown voltage test, when the grease samples break down, the test point tower will immediately drop, the current signal will peak, and at the same time, the electric shock sound will be emitted at the test site. With a constant shear rate of 35 s^{-1} at 25℃ and voltage ranging from 0 to 7000 V. The breakdown voltage of the base grease is 5500 V. With the addition of LCDs, the breakdown voltage significantly decreases by about 600 - 700 V (**Fig. 3b**). Thus, LCDs can reduce the breakdown voltage of base grease, implying the improvement of conductive properties of base grease, which is consistent with the volume resistivity test results.

From the electrorheological tests, the shear stress of the grease samples exhibits significant changes over 10% when the voltage is over 3000 V. Therefore, the viscoelastic properties of grease samples were tested under 0 V and 3000 V at 25℃. The statistical distribution of the shear deformation points of the grease samples obtained from viscoelastic property testing is presented in **Fig. 3c**. The experimental results indicate that adding LCDs enhances the storage modulus and loss modulus of the base grease. The storage modulus corresponds to the elastic properties, while the loss modulus corresponds to its viscous properties [39]. Therefore, the incorporation of LCDs can enhance the viscoelastic properties of the base grease [40, 41]. When applying the voltage, the structural strength of base grease is significantly reduced by over 10% (**Fig. 3d**), indicating that the electric field has a destructive effect on the structure of the base grease [42]. Although the structural strength of the grease with LCDs also reduces under electric field conditions, the degree is smaller than that of the base grease. Especially, the reduced degree for the base grease with 0.25% LCDs is the smallest among the grease samples about 3.56%. That is, the addition of LCDs endows the base grease to resist the effect of the electric field on the structural strength.

According to the test results, base grease shows a bigger breakdown voltage, a poor adaptability and a lower conductivity. LCDs can form a conductive network in grease, reducing the accumulation of charge at the frictional contact site and decreasing the volume resistivity and breakdown voltage. Therefore, under real current-carrying friction conditions, LCDs can weaken the negative lubricating effects caused by them.

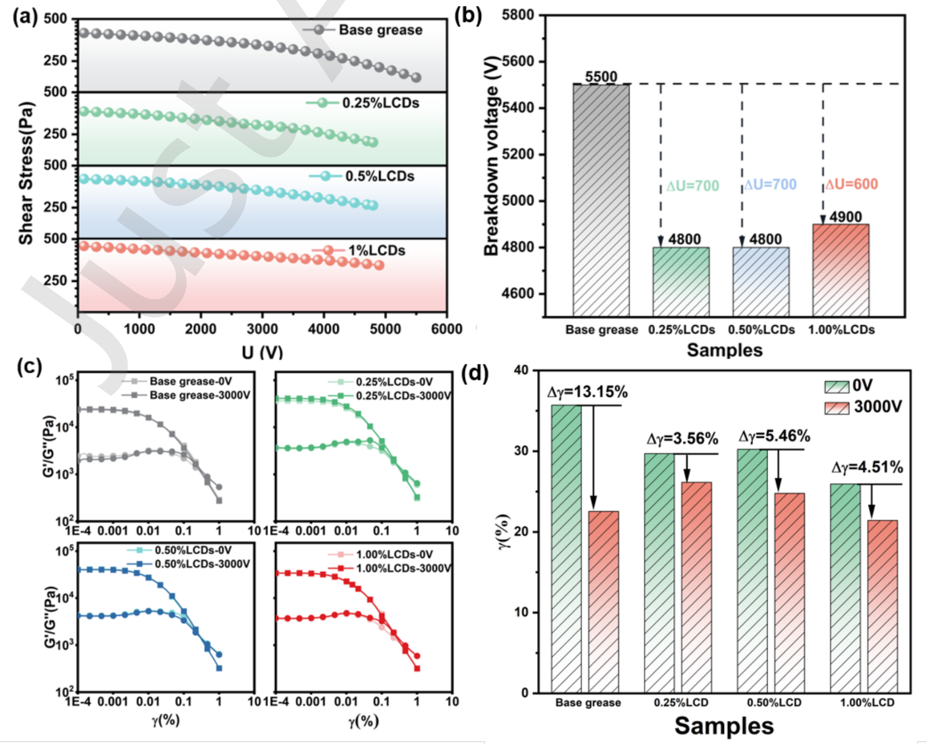

Fig. 3 The results of electrorheological performance about base grease and grease with LCDs

under an electric field (a, b) the breakdown voltage of base grease and grease with different content of LCDs, (c) the viscoelastic properties of grease samples, (d) the shear deformation points of grease samples.

3.4 Worn surface analysis

To investigate the impact of different additive contents on the worn surface, the three-dimensional profile analysis was conducted on the worn surface of the steel ball. The two-dimensional profile in **Fig. 4a** are obtained through the cross profile of the straight three-dimensional graph (**Fig. S2**). In **Fig. 4a**, the base grease shows a deep and wide wear scar. By contrast, the addition of LCDs can significantly reduce the depth and width of the wear scar. Among them, the base grease with 0.25 wt% LCDs exhibits the shallowest wear scar, which is consistent with the friction tests.

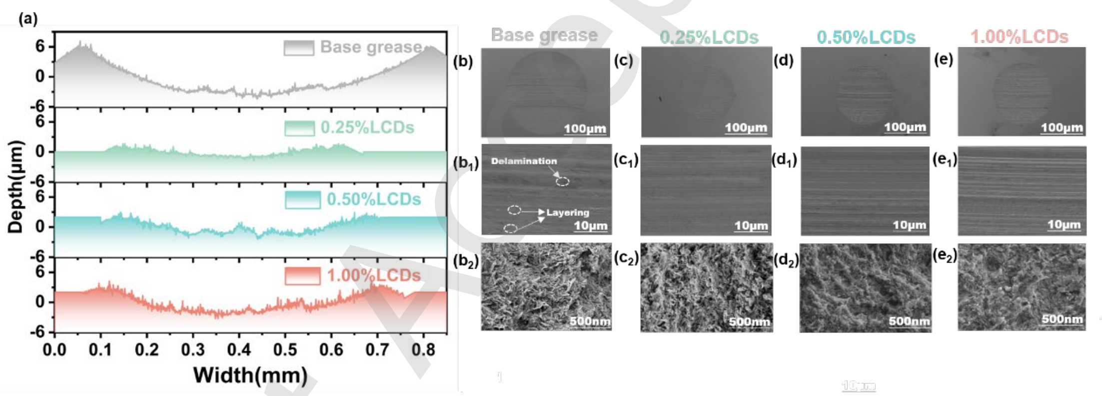

Fig. 4 3D profile (a) and SEM (b-e and b_1-e_1) results of wear surfaces and SEM photos of soap fiber's microstructures (b_2-e_2) for the greases with and without LCDs.

SEM further analyzed the worn surface of steel ball. From **Fig. 4b** and **4b1**, the worn surface of base grease shows a larger wear scar with obvious layering and delamination. After adding LCDs, the size of wear scar is significantly reduced, and the worn surface becomes smoother without obvious layering and delamination. Additionally, with the increased concentration of LCDs, the anti-wear property under current conditions does not improve as expected, but it is still superior to the base grease. This phenomenon might be caused by the no improvement of grease conductivity. To investigate the influences of the addition on the structure of base grease, the soap of grease samples was analyzed by SEM. In **Fig. 4b2**, the base grease soap shows an incompact fiber structure. Notably, an obvious fiber network structure emerges in the grease soap with LCDs (**Fig. 4c2** to **4e2**). Through the EDS mapping of (**Fig. S3**), the elements display a dispersion state in grease soap, indicating that LCDs could dispersed in grease soap stably and cross-linked with them. As the LCDs content increases, the soap fibers become more entangled, which confirms the conjecture of LCDs aggregation.

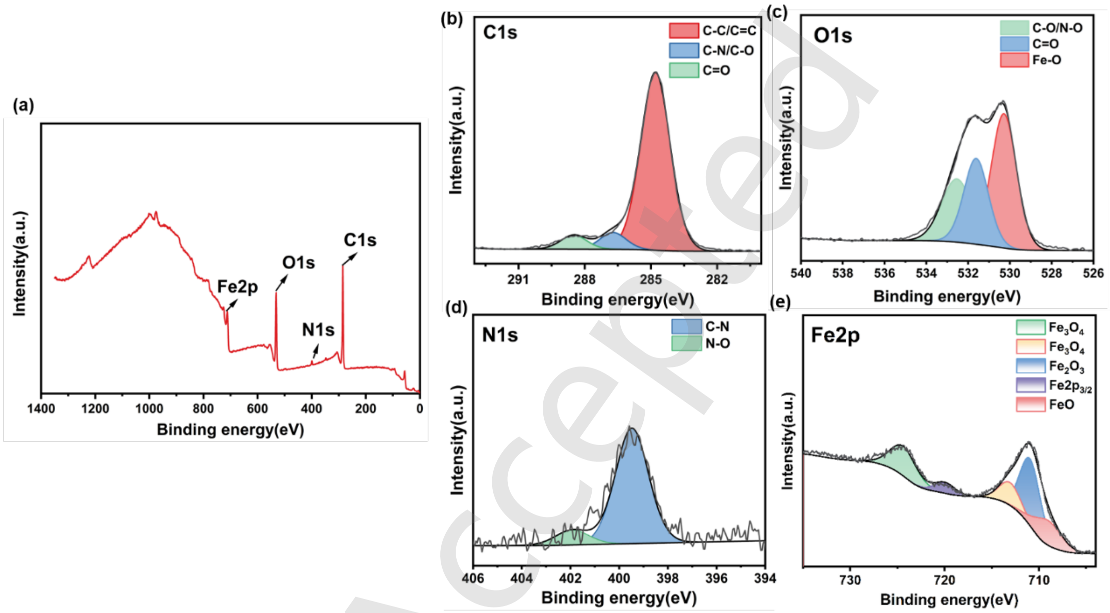

Fig. 5 The XPS spectrum of the worn surface lubricated with base grease with 0.25 wt% of LCDs

To obtain the chemical states of characteristic elements on the worn surface, the XPS analyses were performed. Before of XPS test, the test steel ball was washed through reagents.

For the purpose of comparison, the XPS spectrum of the worn surface lubricated with the base grease is shown in **Fig. S4**. On the worn surface lubricated with the base grease, no additional elements were detected apart from nitrogen, oxygen, and iron. For the worn surface lubricated with the conductive grease, in the full spectrum (**Fig. 5a**), the worn surface contains C, N, O, and Fe elements. In **Fig. 5b**, further analysis of the C 1s spectrum shows that there are three peaks at 284.8, 286.72, and 288.50 eV, which are symbols for C-C/C=C, C-O/C-N, and C=O bonds, respectively. The N 1s spectrum in **Fig. 5c** shows that two peaks at 399.38, and 401.84 eV, which belong to C-N, N-O respectively, all of which are attributed to LCDs. Meanwhile, according to the O 1s and Fe 2p spectra, a peak at 709.06 eV is observed, which can be attributed to FeO[43], the peaks at 713.07 eV and 710.95 eV can be attributed to Fe_2O_3[44], for the peaks at 719 eV and 724 eV are attributed to the formation of Fe_3O_4 and Fe $2p_{2/3}$ on the worn surface[45]. and nitrogen oxides are detected on the worn surface. Furthermore, no nitrogen oxides bonds were detected in XPS characterization of LCDs, indicating that, with the loading current and the friction heat, the tribo-chemical reaction occurs between LCDs and substrate to form tribo-film in the friction process.

3.5 Current-carrying lubrication mechanism

The diagram of the current-carrying lubrication mechanism is shown in **Fig. 6**. On the one hand, based on the electrorheological and volume resistivity test results, the introduction of LCDs enhances the electrical conductivity of lithium-based grease, which prevents the accumulation of heat caused by the higher electrical resistance. Combined with the SEM analyses of the grease soap, LCDs in grease form a fiber network through the permeation effect [33-35]. Meanwhile, the conductive of grease will be improved due to the ionic structure of LCDs, which is consistent with the electrorheological and volume resistivity tests, indicating the formation of conductive network in grease. In addition, liquid state endows LCDs with good dispersibility in grease, which was confirmed by the EDS mapping analyses.

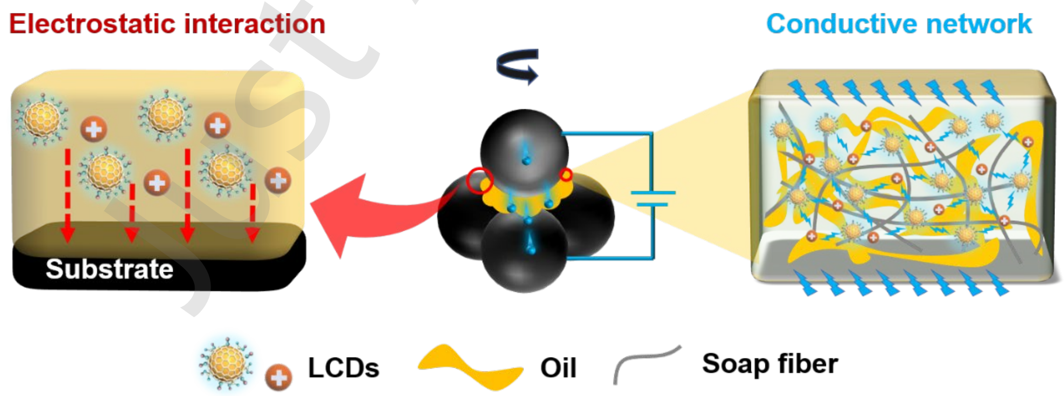

Fig. 6 The mechanism of friction and the conductive diagram of grease with LCDs.

On the other hand, compared with traditional friction, the unique electric current environment enhances the electrostatic interaction on the surface of friction pair. In this case, LCDs containing ionic structure could adsorb on the friction interface easily to form tribo-film in the friction process. From the XPS results, the tribo-film is composed of iron oxides and organic oxidation products, which mainly derive from LCDs. These illustrate that LCDs can still exhibit basic lubricating effects under current-carrying conditions.

In summary, LCDs as conductive additives can be stably dispersed in grease to form conductive network, and play an effective role with lower addition amount. Furthermore, under the dual influence of the ionic structure and current condition, the electrostatic interaction between LCDs and friction interface is further enhanced. These are also the advantages over other materials, such as graphite, CBs, graphene and so on.

4. Conclusion

In this work, LCDs were used as conductive additives in lithium-based grease to study the current-carrying tribological performance. Electrorheological tests, grease soap fiber and worn surface analyses were conducted to study the current-carrying lubrication mechanism. The conclusions are as follows:

1. LCDs can improve conductive performance of lithium-based grease, including reducing breakdown voltage and improving adaptability to electric fields. The grease with 0.25 wt% LCDs exhibits better conductive performance, with the volume resistivity decreasing by one order of magnitude.

2. LCDs improve the current-carrying tribological performance of the lithium-based grease. In particular, when at the lower concentration (0.25 wt%), LCDs still exhibit the effective current-carrying lubrication properties. In addition, LCDs reduce the heat generated during the current-carrying friction process, showing a lower ΔT.

3. LCDs with good dispersibility and ionic structure form a conductive network in grease. The conductive network enhances the current transmission capacity, decreasing the heat accumulation caused by the thermal effect of current. The electrostatic interaction between LCDs and the friction interface promotes the adsorption of LCDs, allowing them to exhibit lubricating properties and prevent current-carrying friction.

Data availability

All research data supporting this publication are directly available within this publication.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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