Study on the effect of mechanical shearing on the microstructure and performance of lithium complex grease

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Study on the effect of mechanical shearing on the microstructure and performance of lithium complex grease

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Abstract:

This paper focuses on the effect of mechanical shearing on the structure and properties of complex lithium greases with or without zinc dialkyl dithiophosphate (ZDDP). The base grease and grease with additives were first mechanically sheared by a roller shear tester, and then the tribological properties, rheological properties, microstructure changes and noise properties of the samples were tested by a four-ball tester, rheometer, field emission scanning electron microscope and Bequiet+ noise tester, respectively. The results indicated that mechanical shear significantly reduced the mechanical stability and colloidal stability of the lithium grease, and the tribological properties of the two greases (with or without ZDDP) showed opposite trends. The rheological performance test showed that the structural strength of the grease was significantly reduced and the microstructure became loose after shearing. In addition, the grease

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containing ZDDP showed a slight decrease in noise compared to the base grease due to the favorable reduction of the intermediate frequency of the complex lithium grease. This study provided theoretical guidance for the precise design and performance optimization of low noise greases.
Keywords: Lithium complex grease; ZDDP; Friction; Rheology; Noise property; Micro-structure

1. Introduction

Grease is one of the major bearing components and the performance of bearing is strongly determined by grease [1]. Grease is subjected to a variety of environmental factors during operation, such as extrusion and shearing. Changes in these factors will alter the performance of the grease and may also greatly affect the microstructure of the soap fibers. Most of the functional characteristics of greases are related to their mechanical shear stability and the ability to flow under mechanical stress, which in turn affects their service life [2]. Therefore, it is significantly important to study the overall performance changes of grease under shearing.

Research on the performance change of grease after mechanical shearing has been widely considered by researchers, and many theoretical models have been established through simulated shear experiments. He et al. [3] studied the influence of the mechanical stability of bentonite grease, and the results showed that the layered structure of bentonite grease that was not covered by the organic layer dissociated under the influence of shear and lost its thickening ability. Lundberg and Hoglund [4] summarized the importance and effectiveness of various mechanical stability experiments by studying grease samples in wheels. They found that γ values correlate well with the mechanical stability in service. Furthermore, Kuhn [5,6] defined the rheological energy density model and mechanical dissipation function to represent the structural degradation of grease and proposed applying the concept of entropy to mechanical degradation. Moore and Cravat [7] proposed an exponential model in which the decreasing rate of the consistency of grease is proportional to the structural destruction rate of the thickener by studying the structural destruction rate of the aging soap-based grease thickener. Czarny [8] established an empirical equation to describe the decrease in grease viscosity in terms of the number of shear cycles. Based on Czarny's work, Plint et al. [9] proposed a half-life parameter to describe the aging of grease in lubrication workers. Based on previous theories, Rezasoltani and Khonsari [10] used an improved Couette aging machine and rheometer to conduct long-term shear experiments on lithium grease and established a shear life model. It was found that there was a linear relationship between energy input and grease performance, which was independent of the applied shear rate and grease temperature. Furthermore, Zhou et al. [11] compared the film thickness of grease before and after mechanically aging in a shielded deep groove ball bearing. Results showed that film thickness after long time was dependent on oil viscosity, grease bleed, and the shear degradation of microstructure. Moreover, Zhou et al. [12] proposed a master curve for lithium and polyurea grease with a fibrous structure under shear degradation, in order to describe the influence of shear and temperature on the mechanical aging of greases.

Previous studies on the influence of mechanical shear on grease are mostly focused on the establishment of theoretical models or describing the influence on the shear life, but there is a lack of research on the properties, microstructure changes, and service behaviors of grease under mechanical shear, and the influence of additives in shear is also not taken into consideration. In particular, the demand for high stability and low noise performance of mechanical devices is increasing. Therefore, the mechanical shear of complex lithium base grease with or without the anti-wear additive zinc dialkyl dithiophosphate (ZDDP) was simulated by a roller experiment in this paper. The influence of shear on microstructure, rheological and tribological performance as well as noise property of grease were studied, which could provide a theoretical basis for further study on the influence of mechanical shear on grease.

2. Materials and Experiments

2.1 Preparation of complex lithium base grease

PAO40 (Mobil Corporation), 150BS (Thailand IRPC Co., Ltd.), AN15 (King Industries, Inc.), 900N (Jingmen Petrochemical Co., Ltd.) were added into a clean reactor and heated to 80 °C. Then, 12-hydroxystearic acid (Dongying Shunli Chemical Co., Ltd.) with accurate quantity was added into the base oil. When the temperature rose to 95°C, the preheated 1/3 lithium hydroxide aqueous solution was slowly added within 20 min, and the reaction was carried out at a constant temperature for 90 min. Afterward, azelaic acid (Sichuan Xipu Chemical Co., Ltd.) was added and kept for 15 minutes. When the temperature in the kettle rose to 100 °C, the remaining lithium hydroxide aqueous solution was slowly added and reacted for 90 min. After dehydration at 135 °C for 30 min, the mixture was heated to 206 °C for refining and kept for 30 min, and then the complex lithium grease was obtained after cooling down. The basic properties of grease are shown in Table 1.

The complex lithium grease is abbreviated as BG, and grease mixed with 2 wt.% ZDDP (code T202, Wuxi Nanfang Petroleum Additives Co., Ltd.) is abbreviated as BGT202. Then, BG and BGT202 were placed in a constant temperature drying oven with a set temperature of 50 °C for 30 min. After drying, all the samples were stirred and ground 3 times using a three-roll mill.

Table 1 Typical properties of synthesized complex lithium grease

Properties	Specification
Thickener	Lithium 12-hydroxystearate, dilithium azelate
Type of base oil	PAO40、150BS、AN15、900N
Dropping point(°C)	330
Cone penetration(0.1 mm)	253
Bleeding test (%)	4.09

2.2 Shearing process

The shearing process was performed using the grease drum testing machine (made by North Dalian Analytical Instruments, No. BF102A). 50g of BG and BGT202 were evenly smeared on the inner wall of the cleaned drum. The speed was set at 165±5 rad/s, and the temperature was set at 80±1 °C. The shearing time was set as 0 h, 2 h, 4 h, 8 h, and 24 h, respectively.

2.3 Basic performance test

The 1/4 cone penetration test according to GB/T 269 was used to evaluate the mechanical stability of BG and BGT202 after shearing treatment at different time intervals. The steel mesh oil separation test according to SH/T 0324 was used to describe the colloidal stability of different grease samples.

2.4 Tribological performance test

A lever-type four-ball testing machine (Xiamen Tenkey, MS-10A) was used to test the variation in the friction coefficient and wear scar diameter under boundary lubrication condition. The experiments were conducted according to SH/T 0204 with a load of 392 N, a rotating speed of 1200 rad/min, a testing temperature of 75±1 °C, and a test duration of 60 min.

2.5 Rheological property test

The viscoelastic and apparent viscosity changes of BG and BGT202 after shearing for different time intervals were measured by a rotary rheometer (Anton Par, MCR 302). The Storage Modulus (G') and Loss Modulus (G'') curves were obtained by plate oscillation mode with plate-to-plate sandblasted parallel disks (the diameter was 24.958 mm and the gap was 1 mm). The strain was set from 0.01% to 100%, and the testing temperature was 25 °C. The apparent viscosity was measured at a shear rate of 300 rad/min for 300 s at 80 °C.

2.6 Microscopic structure observation

Field emission scanning electron microscopy (FE-SEM, OPTON NTS Limited, Merlin Compact) was used to observe the changes in the microstructure of the lubricant samples after shearing for different time length. The sample was dissolved in petroleum ether, and the base oil was removed by centrifugation. One drop of the solution containing soap fiber was placed on the carbon film copper net and dried at room temperature. After spraying gold, the microstructure of the thickener was observed and photographed by FE-SEM [13].

2.7 Noise performance test

The noise performance of grease after shearing for different time intervals was tested with a Bequiet+ grease low noise tester of SKF Company in Sweden, and the bearing grade was BY-608/VQ607. The spindle speed was set as 1800 r/min, and the axial load was 30 N. Grease was injected for 5.00 s and measured for 3.20 s.

3. Results and discussion

3.1 Basic performance test

The 1/4 cone penetration values of BG and BGT202 in the drum with different shearing time length are summarized in Fig. 1(a). The cone penetration of BGT202 is significantly higher than that of BG which is not sheared. The addition of T202 increases the cone penetration of base grease, which may be because T202 is liquid and changes the consistency of the entire system. When the shear time gradually increases from 2 h to 24 h, the cone penetration shows a trend of gradual increase. The increment in cone penetration of BG is the largest after sheared for 2 h, while the increment of shearing for 4 h, 8 h, and 24 h becomes smaller. The trend of cone penetration of BGT202 was consistent with that of BG, and even decreases slightly from 8 h to 24 h. The influence of shear time on the shear stability of grease does not increase linearly. Therefore, it can be concluded that the consistency of grease decreases greatly in the initial shear stage and the decreasing amplitude slows down when the shear time extends.

Fig. 1(b) shows the variation in the oil separation ratio of BG and BGT202 after shearing for different hours. The oil bleeding of BGT202 without shear is greater than that of BG. With the increase of shear time, the oil bleeding of BG and BGT202 increases gradually, and BGT202 is higher at the same shear time. The results show that mechanical shearing can attenuate the colloidal stability of grease, and the addition of T202 will promote the attenuation.

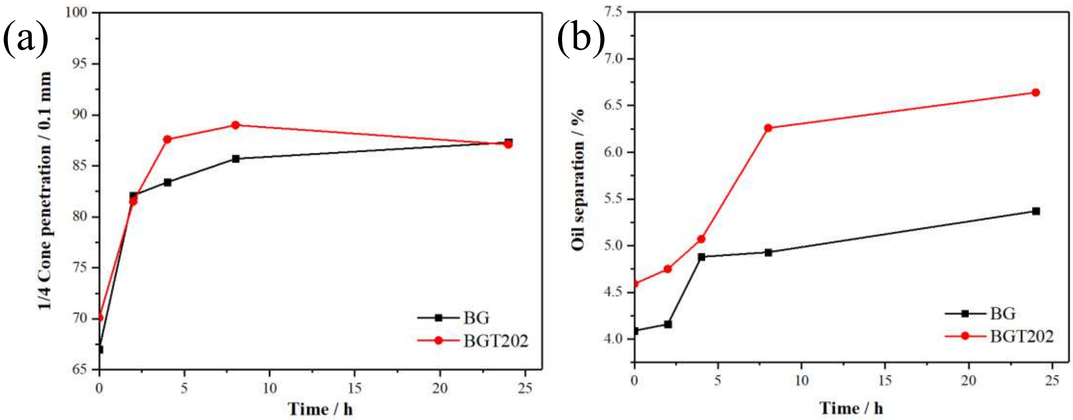

Fig.1 Variation in 1/4 cone penetration (a) and oil separation ratio (b) of BG and BGT202 after shearing for different hours

3.2 Tribological performance test

The primary function of grease is lubrication, and the evaluation of friction and wear is an important means to characterize the lubrication performance. Fig. 2 shows the friction coefficient curves of BG and BGT202 after shearing for different hours. The initial friction coefficient of the BG without shear exceeds 0.13 and then stabilizes at approximately 0.085, but there are many convex peaks with large fluctuations throughout the test. Compared with BG, the friction coefficient curves of BG-2, BG-4 and BG-8 are gentle and no large convex peak appears. As the average friction coefficient decreases with the increase of shear time, the friction-reducing effect is improved to varying degrees.

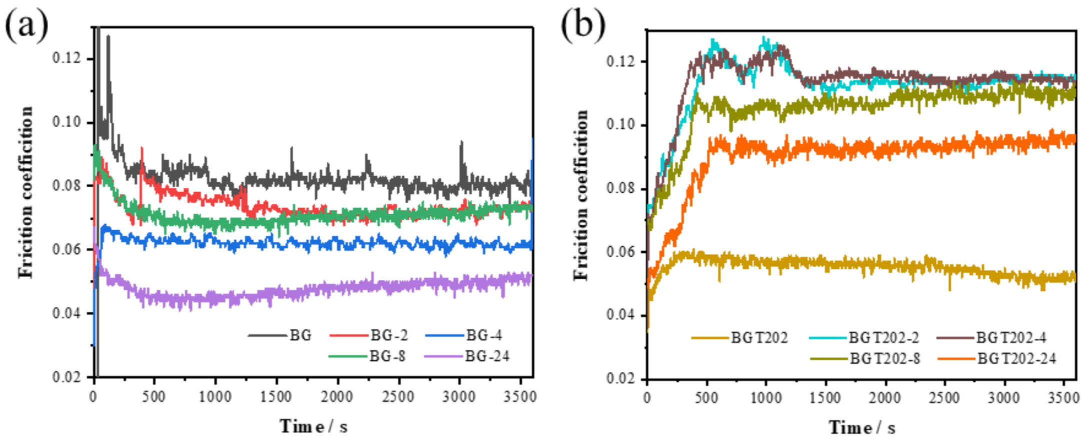

Fig. 2 Friction coefficient of greases after shearing for different hours (a: BG, b: BGT202)

The variation trend of the friction coefficient of BGT202 after shearing for different hours is quite different from that of BG. BGT202, whose friction coefficient is lower than 0.06 after the first 500 s, shows excellent friction-reducing performance. However, the curves of BGT202-2, BGT202-4 and BGT202-8 stabilize at approximately 0.11, and that of BGT202-24 is around 0.09. Therefore, it shows that the friction-reducing effect of BGT202 is weakened after shearing, which is entirely different with that of BG.

Fig. 3 shows the variation of wear scar diameter of BG and BGT202 after shearing for different hours. The diameter of the BG is 0.616 mm and then decreases gradually with increasing shearing time. When shearing for 24 h, the wear scar diameter reaches 0.441 mm. In contrast, the diameter of BGT202 is 0.429 mm and increases gradually with shearing time. When shearing for 24 h, the diameter reaches 0.498 mm, with an increased rate of more than 16%. The above experimental results indicate that mechanical shearing is beneficial to the lubricating effect of BG but not conducive to the anti-friction and anti-wear performance of grease containing T202. However, the author believes that the improvement of the tribological properties of BG by mechanical shear is based on the premise of sacrificing the colloidal and mechanical stability of grease, thus improving the lubricating property by reducing the consistency of grease and increasing the oil separation. After adding T202 to the base grease, the lubricating film formed by the reaction between active sulfur, phosphorus elements generated by high-temperature decomposition of T202 and iron elements on the metal surface adheres to the surface of the friction contact area, thereby reducing the coefficient of friction and wear scar diameter [14]. T202 in lubricating grease is gradually depleted with the increase of shear time, resulting in a gradual decrease in anti-friction and anti-wear properties. The reason for the decrease in the friction coefficient after shearing for 24 hours is the same as that of the base grease after shearing. That is, the decrease in its consistency and increase in oil separation improve the lubrication state of the friction pair and become a leading factor in the decrease in the friction coefficient. It is worth noting that although the substantial increase in the consistency and oil separation rate of grease improves the friction performance of the base grease to a certain extent, it easily leads to the loss of grease from the friction pair.

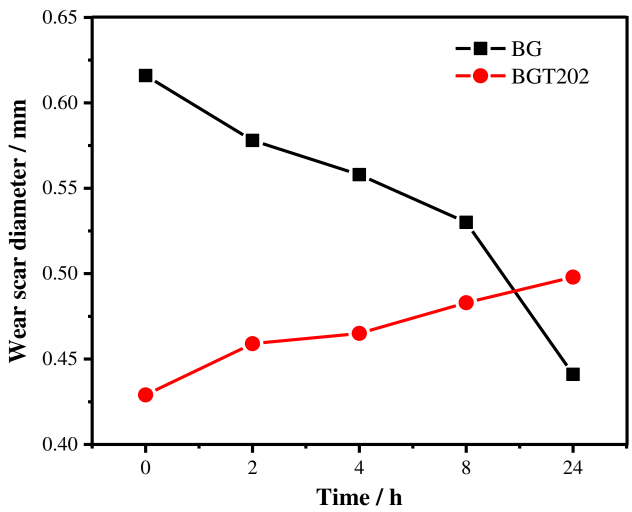

Fig. 3 Wear scar diameters of BG and BGT202 after shearing for different hours

3.3 Rheological property test

The rheological properties of BG and BGT202 after shearing for different time length were investigated. The storage modulus (G') represents the elastic deformation energy stored by the grease to maintain the gel properties, while the loss modulus (G'')

represents the energy consumed by the viscous flow of the grease[15]. In the linear viscoelastic region, the G' and G'' curves are stable, and the soap-fiber structure of grease still maintains its initial shape. With the increase of strain into the nonlinear viscoelastic region, G' and G'' begin to decrease sharply, and the grease gradually changes from solid-state ($G' > G''$) to liquid-state ($G' < G''$). In this process, the intersection point of the storage modulus and loss modulus ($G' = G''$) is called the structural conversion point. Therefore, the shear stress value (τ_f) corresponding to the structural transformation point can be used to characterize the structural strength of grease soap fibers [16, 17].

Fig. 4 shows the G', G'' and shear stress of BG and BGT202 after shearing for different hours as the strain increases. As it is shown, G' and G'' of BG after shearing for 2 h, 4 h and 8 h are slightly reduced compared with that without shearing. After shearing for 24 hours, G' and G'' of BG decrease significantly, and the intersection point of G' and G'' moves to the lower strain. In contrast, the curves of G' and G'' of BGT202 change little after shearing for 24 h. The viscoelastic properties of BG and BGT202 are mainly influenced by long-time mechanical shearing, while short-time shear shows little influence. Compared with BG sheared for different hours, the curves of BGT202 after shearing are more stable, which might be attributed to the enhancement of the molecular force between the complex lithium grease soap systems by T202. The change in microstructure of greases also could explain the difference of rheological property between BG and BGT202, which will be discussed in the next chapter. In addition, the shear stress at the transformation point of BG and BGT202 after shearing show an obvious decreasing trend with the increase of shearing time, indicating that the structural strength of grease soap fiber is attenuated after shearing.

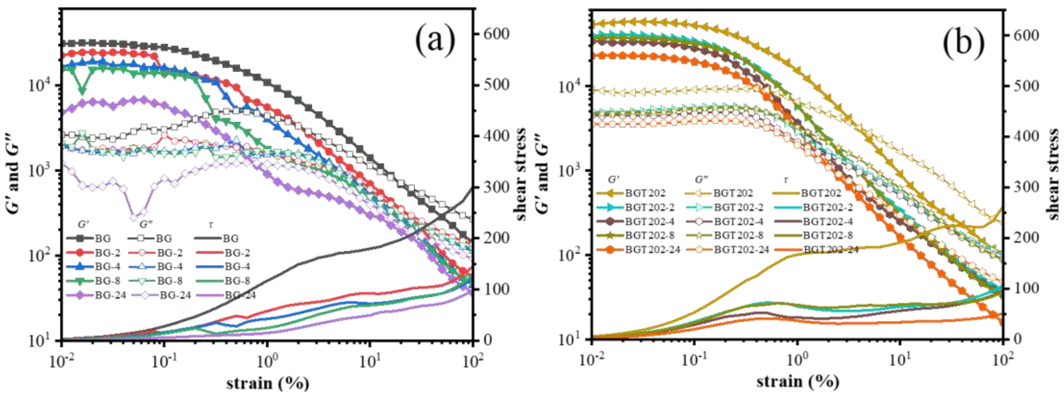

Fig. 4 Storage modulus (G'), loss modulus (G'') and shear stress (τ) of the complex lithium greases as a function of amplitude (a: BG, b: BGT202)

Fig. 5 shows the curves of the apparent viscosity of BG and BGT202 samples after shearing for different hours at a constant shear rate of 300 rad/min for 300 s. It can be seen that the viscosity of BG and BGT202 is high without shearing, then decreases slowly with increasing time and tends to a constant value. For BG, the apparent viscosity curves at 2 h, 4 h, 8 h and 24 h are significantly lower than those without shearing. The curves of greases sheared for 2 h, 4 h and 8 h only slightly decrease, and that of 24 h shear shows a great decrease compared with the other samples. For BGT202, the curve of grease sheared for 2 h showed a significant decrease compared with the grease without shearing. The curves of grease for 4 h and 8 h are very similar and lie between greases of 2 h and 24 h. In general, the apparent viscosity decreases with the prolongation of shear time, but the magnitude is different. There is a limit to the influence of mechanical shear on the apparent viscosity of grease. The viscosity decreases when increasing shearing time, but the decreasing amplitude gradually becomes smaller. At the same time, the decrease in structural strength and apparent viscosity in the rheological experiments of BG and BGT202 is in good agreement with the variation in mechanical stability as discussed above.

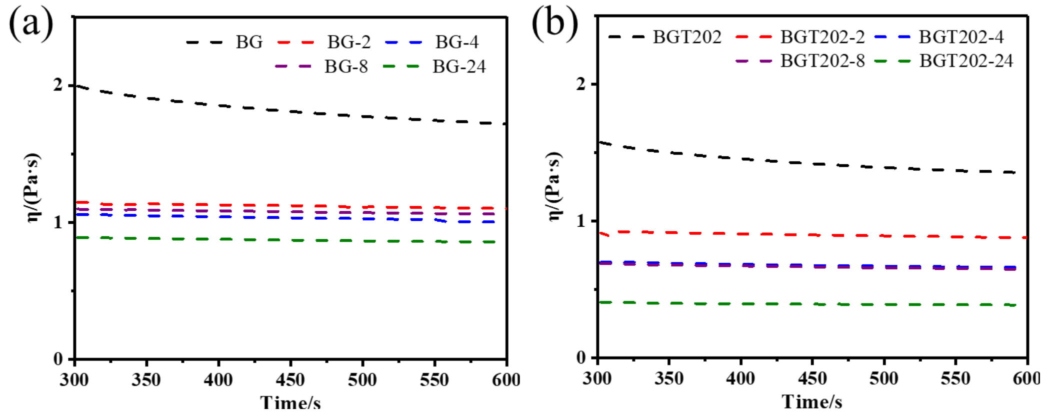

Fig. 5 Apparent viscosity changes of BG and BGT202 samples measured at a constant shear rate of 300 rad/min for 300 s

3.4 Microstructure of Greases

Previous studies have shown that there is a strong relationship between the tribological and rheological properties of grease and the change in its microstructure [16-18]. Herein, FE-SEM was used to observe the changes in terms of the microscopic morphology of BG and BGT202 soap fibers before and after shearing, and the results were displayed in Fig. 6 and Fig. 7.

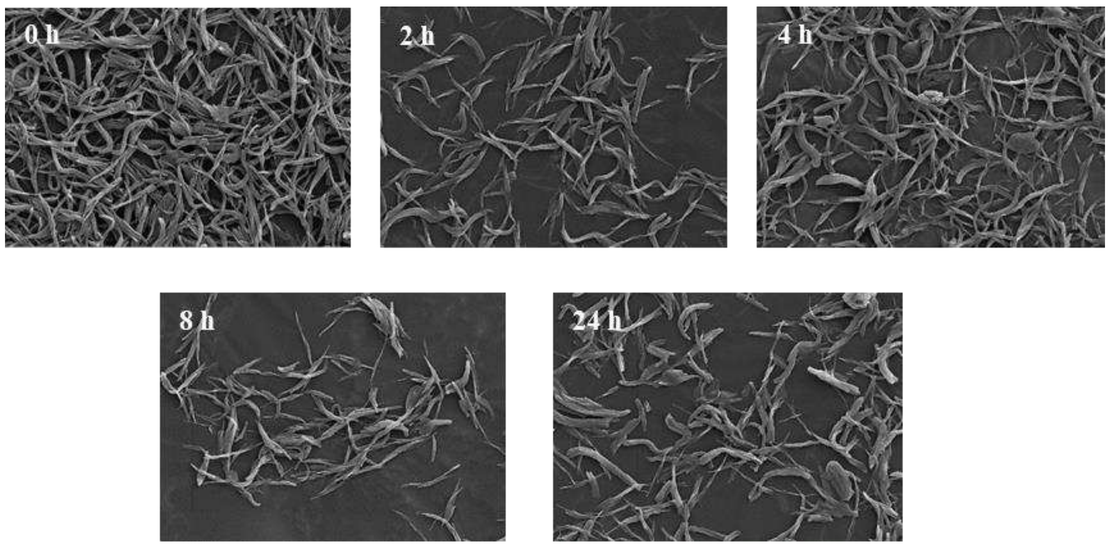

Fig. 6 SEM microstructure images of BG before and after shearing

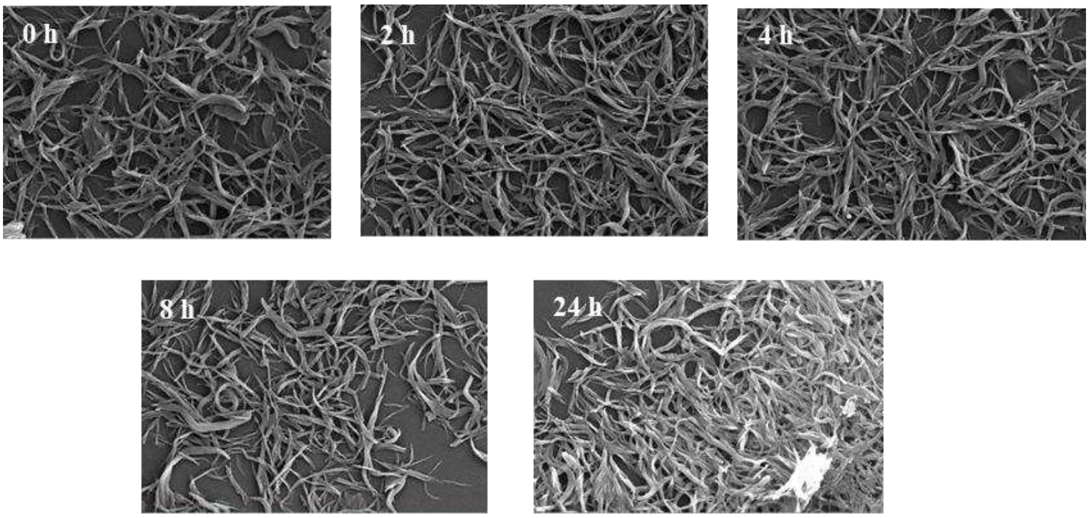

Fig. 7 SEM microstructure images of BGT202 before and after shearing

Fig. 6 shows the SEM images of soap fiber taken by BG after shearing for 0 h, 2 h, 4 h, 8 h and 24 h. When BG is not sheared, the soap fiber is relatively dense and uniform in thickness. After shearing for 2 h, 4 h, 8 h and 24 h, different amounts of short fibers are generated in each sample as shown in the figure, and some chain breaks appear. After shearing for 8 h and 24 h, a few lumpy fibers appear in the soap fiber masses, and the structure becomes looser. The changes in BG soap fiber after shearing are macroscopically reflected in the deterioration of the bundle oil capacity of grease and the decrease in colloidal stability and mechanical stability. The change in microstructure can also explain the deterioration of rheological properties to some extent. When the structure of soap fiber becomes loose, the shear stress corresponding to the transformation point of the structure in the rheological diagram will decrease correspondingly, indicating the decrease of the structural strength of grease. Furthermore, Fig. 7 shows the SEM image of the soap fiber microstructure of BGT202 after shearing for 0 h, 2 h, 4 h, 8 h and 24 h. The microstructure of BGT202 soap fiber after different shearing time length is different from that of BG. Compared with the unsheared sample, only a few soap fibers of BGT202 appear to bulge after shearing for different time length, and the overall fiber network maintains relatively well, which probably results in the relatively stable viscoelastic energy curve of BGT202 in the rheological diagram after shearing. Moreover, the soap fiber of BGT202 after shearing has no significant change as shown in the SEM images, but the shear stress decreases significantly after shearing, which might be due to the attenuation of molecular force to some extent after shearing.

3.5 Noise Property

Bequiet+ test transforms mechanical vibration and the pulses into audible sound using a loudspeaker and gives a qualitative indication of the situation in the high-speed operating bearing system. This study presents vibration velocity data of medium and high frequencies for BG and BGT202, both of which reflect the noise performance of the grease, as shown in Fig. 8. As for BG, the data of medium frequency increases after shearing for different time length, and the vibration velocity is relatively high for greases sheared for 8 h and 24 h, indicating that the noise reduction performance of BG is weakened after shearing. In contrast, the medium frequency of BGT202 remained relatively stable after shearing for different time length.

In contrast, both BG and BGT202 undergo significant changes in the high-frequency band after shearing for different time length. Compared with BGT202, the BG vibration peak speed in the high-frequency band is more dispersed, and its noise reduction performance is worse, indicating the addition of T202 is conducive to noise reduction of grease. The improvement in noise property by T202 might be related to the strengthening effect of additive molecules on the fiber structure of complex lithium grease, and no obvious damage to the microstructure of BGT202 after shear can also prove this view. However, the performance of BG and BGT202 in the high-frequency band is relatively poor, and the vibration velocity peaks before and after shear show great fluctuations. This is mainly because, although T202 is helpful to the noise reduction of the grease in the medium-frequency band, the low noise performance of base grease (related to raw materials and production process) is the dominant factor when reaching the high-frequency band [19, 20].

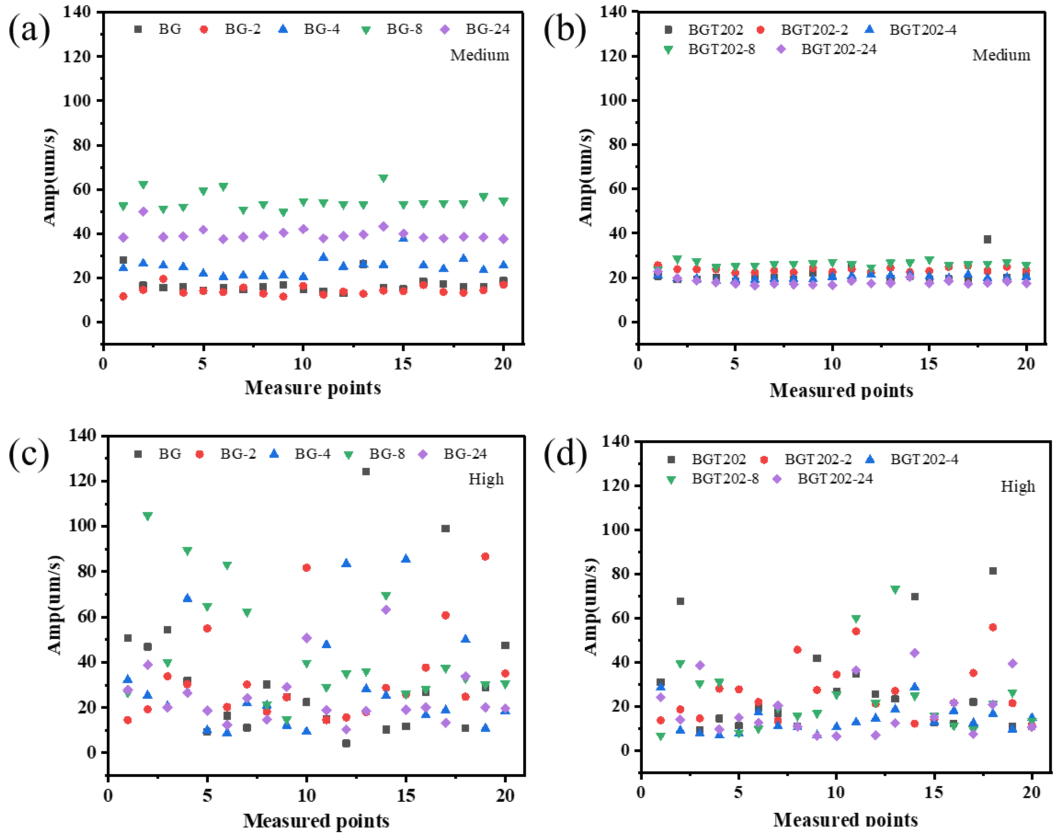

Fig. 8 Comparison of medium and high frequencies of noise for BG (a, c) and BGT202 (b, d) after shearing for different hours

4. Conclusions

The basic properties, friction and wear properties, rheological properties, microstructure and noise properties of complex lithium grease with and without 2 wt.% T202 were studied after shearing for different hours. The following conclusions can be drawn:

a. Complex lithium grease under mechanical shearing will become thinning, resulting in an increase in cone penetration and a decrease in mechanical stability. At the same time, the colloid stability of the composite lithium grease decreases after shearing, indicating by an increase in the oil separation rate.

b. The base oil of grease is released after shear thinning and generates an oil film to separate the surface of the friction pair, which is conducive to lubrication. As T202 is gradually consumed in the shear process, the anti-wear effect of BGT202 is slightly weakened.

c. After mechanical shearing for different time length, the structural strength of BG and BGT202 can be found to decrease obviously from the rheological test. The degree of microstructure entanglement in BG significantly reduces but not in BGT202.

d. The soap-fiber structure of BGT202 is more stable than that of BG, so the noise reduction performance is better after shearing. Mechanical shearing is beneficial for BGT202 to reduce the peak vibration velocity in the medium-frequency band but not significantly for the high-frequency band.

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Declarations

Conflict of interest The authors have no relevant fnancial or non-financial interests to disclose.