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Effect of Heat Treatment on Lubricating Properties of Lithium Lubricating Grease

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Abstract

Owing to potential oxidation and thermal degradation of lithium lubricating grease when the grease was heated to improve its fluidity in the pumping process, fresh NLGI 3 lithium lubricating grease samples were heated to obtain experimental samples to simulate the static thermal degradation by using drying oven. The microstructure and infrared spectra were studied by using field emission scanning electron microscopy (FESEM) and Fourier transform infrared spectroscopy (FTIR) techniques respectively. The rheological properties were estimated by rotational rheometer. And the tribological properties were evaluated on a four-ball testing machine. At last, the action mechanisms of the effect of heat treatment on tribological properties of lithium grease were discussed. The experimental results showed that the lithium lubricating grease samples exhibited better antifriction and antiwear properties after heat treatment at 120 °C, which is not obvious during 0 h-4 h, while the coefficient friction and extreme pressure progressively decreased during 4 h-24 h. Meanwhile, oxidative and thermal degradation do not proceed after heat treatment for 0 h-24 h according to the results of FESEM and FTIR. Finally, it was confirmed that the variation of physical entanglement, which was caused by a short time heat treatment, would vary the tribological properties of lithium lubricating grease.

Keywords: Lubricating grease; heat treatment; tribological properties; microstructure; infrared spectra; rheological properties

Introduction

Lubricating grease, a semi-solid colloidal dispersion system, is consisted of thickener and base oil, and displays high-viscosity and weak fluidity properties.^{1,2} Therefore, a high pipe resistance would be shown when the grease is delivered by the centralized lubrication system, which is a disadvantage for the centralized lubrication system application. At present, only parts of lubricating greases, which have a better fluidity, are delivered by centralized lubrication system, such as NLGI 1, 0, 00 lubricating greases. However, NLGI 2 and NLGI 3 lubricating greases, which are widely used in the industrial application, are difficult to be delivered by centralized lubrication system due to their weak fluidity at room temperature. Li et al.³ detected that the grease displays weak fluidity in the experimental channel and certain distance is required to let the flow develop fully. Delgado et al.⁴ found that a larger pressure drop appeared when the grease was delivered by pipes. Moreover, lubricating greases manifest excellent viscosity-temperature characteristic and thixotropic property.^{5,6} The fluidity of lubricating grease increases with the increase of temperature. And the fluidity can be fully recovered when the temperature drops to room temperature. Therefore, the deliver resistance can be decreased through heating the grease, and thus to improve the pumpability in the centralized lubricating system.

However, oxidative and thermal degradation proceed when the lubricating grease was subjected to high temperatures, which would shorten the grease lubricating life.⁷ Cann et al.^{8,9} detected that the grease degradation initiated at the motion state of bearings and the temperature are important reasons for degradation increase. Yu et al.¹⁰ found that the poor lubrication which is resulted in the thermal degradation of lubricating grease due to high service temperature is one of the most important reasons for serious local wear in fatigue failure analysis of roller bearing.

Gonçalves et al.¹¹ got that the thickener matrix structure varied after thermal ageing.

Therefore, the fluidity and pumpability of lithium lubricating grease would be improved through heating the grease, which is highly possible to result in oxidative and thermal degradation of the lubricating grease. The lubrication performance of lubricating grease is weakened due to the oxidative and thermal degradation. Wu et al.¹² found that the hydroperoxides was formed during the initial oxidation process, which could deteriorate lubrication performance of di-2-ethylhexyl sebacate (DEHS). Kreivaitis et al.¹³ investigated the effect of thermal oxidation on tribological properties of rapeseed oil and detected that lubrication performance decreases with the process of oxidation due to structural changes in oil. According to the research of Haseeb et al.¹⁴, the biodiesel oxidizes with the increase of test temperature. In addition, the content of free water increases, and the antifriction and antiwear properties are improved as well. Furthermore, tribological properties of lubricating grease are generally used to evaluate its lubrication performance^{15,16}. Therefore, the variation of grease lubrication performance caused by heat treatment could be described via its tribological properties.

The objective of the present investigation was to provide the effect of heat treatment on lubricating properties of NLGI 3 lithium lubricating grease. And in this investigation, 120 °C, which is the highest temperature in standard ball bearings test^{11,17,18}, was selected as the experimental temperature. The microstructure and infrared spectra of lithium lubricating grease were investigated by FESEM and FTIR techniques, and detailed variations were discussed. The strength of microstructural network was estimated through rheological properties by using rotational rheometer. The tribological properties were measured by four-ball testing machine, and the action mechanisms were discussed as well. The research results could detect the effect of heat treatment on lithium

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lubricating grease and provide applicable parameters for pumping NLGI 3 lithium lubricating grease.

Table 1 Main components and technical data for the lubricating greases studied

Thickener type	Thickener %(w/w)	Base Fluid	Lubricating oil viscosity at 40 °C ASTM D-445 (mm ² .s ⁻¹)	Dropping point ASTM D-566 (°C)	Consistency NLGI Grade	Worked penetration ASTM D-217 (dmm)
Lithium 12-hydroxystearate	12.9	Mineral	100	200	3	232

Experiments

Materials

Sample of NLGI 3 lithium lubricating grease was produced by Sinopec Lubricating Oil Co., LTD (China). The main components and technical data of the lubricating grease samples were listed in Table 1.

Sample preparation

The fresh grease with a weight of 10 g was spread in 250 ml glass beakers (thickness was about 1-2 mm) for four groups. And they are kept at atmospheric pressure at 120 °C for 2 h, 4 h, 8 h and 24 h by using drying oven respectively. At the same time, the mass loss of each sample was recorded. In order to limit the influence of water vapor on weight loss experiment in this paper, the samples had been weighted for several times and the final weight was obtained until when the weight of samples did not add up any longer. Besides, there are four groups of fresh grease, each of which has one glass tube with 2 g grease spread evenly. After heat treatment for 2 h, 4 h, 8 h and 24 h, the color of each sample was recorded respectively.

Property tests

The mass loss rate after heat treatment was computed according to formula:

$$\Delta W_T = \frac{M_0 - M_T}{M_0} \times 100\% \quad (1)$$

Where ΔW_T expresses the mass loss rate after heat treatment for T h; M_0 is the original sample weight (g); M_T is the final weight of the sample after heat treatment for T h (g).

The microstructural characterisation of lubricating greases was carried out by means of FESEM (Supera 55 from Zeiss, Germany) at 10 kV. Grease samples were prepared by extracting the oil with *n*-heptane in several batches. Afterwards, the sample was dried at room temperature. Finally, the samples were coated with gold.

FTIR measurements were carried out in a VERTEX 80v FTIR spectrometer (Bruker, Germany) in transmission mode. Greases were placed on a silicon support in order to realize infrared spectra.

Rheological measurements were performed under controlled stress and in a controlled shear rheometer (Physica MCR302 from Anton Paar, Germany) at the temperature of 25 °C by using a plate-plate geometry (50 mm diameter, 1 mm gap). Small-amplitude oscillatory shear tests were carried out in a frequency range between 10⁻¹ s⁻¹ and 10² s⁻¹. Apparent viscosity tests were performed at a constant shear rate (100 s⁻¹)

for 1000 s.

A four-ball testing machine with speed of 1450 r/min was used (MMW-1 from Jinnan testing machine plant, China). The anti-friction and antiwear tests were conducted under 300 N for 30 min. The maximum non-seizure load (P_B value) was determined following the GB 12583-1998 standard, which is similar to ASTM D2783. A ball of 12.7 mm diameter made of GCr 15 with a 61-64 HRC was used. Each test was repeated for three times to minimize data scattering. At the end of each test, the three lower balls were cleaned in petroleum ether and acetone respectively. These cleaned balls were available for wear scar diameter (WSD) measurement. The mean WSD on the three lower balls was measured with an eyesight microscope to an accurate reading of 0.01 mm. Then the arithmetical average WSD of the three identical tests was calculated as the WSD.

Results and discussion

Mass loss rate and color

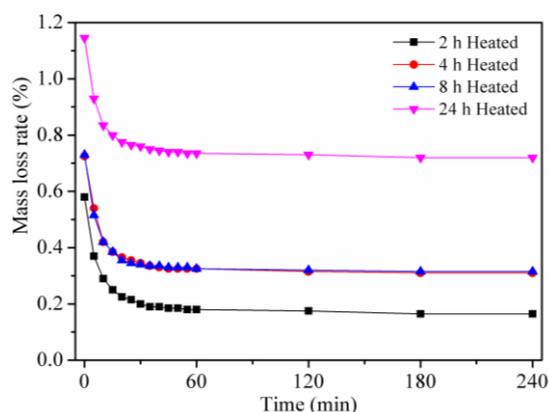


Fig. 1 The variation of Mass loss rate after heat treatment at different times

Fig.1 shows the variation of the mass loss rate for grease samples as a function of placed time after heat treatment of 120 °C for 2 h, 4 h, 8 h and 24 h respectively. It is found that the mass loss rate decreases along with the prolongation of placed time, and tends to a stable value at last. This phenomenon is mainly attributed to the hydrophilic groups in lithium lubricating grease. When the fresh grease samples were placed in drying oven at 120 °C, the moisture combined with hydrophilic groups would be evaporated. However, the hydrophilic groups would absorb the moisture quickly if the samples were placed again in the air. Therefore, the final stable value should be used to investigate the mass loss rate, which could keep both evaporated and absorbed moisture balance and eliminate the effect of hydrophilic groups. As can be observed

in Fig.1, the mass loss rate generally increases with the increase of heat treatment time. However, the mass loss rate would basically be the same during 4 h to 8 h, and increases obviously during 8 h to 24 h.

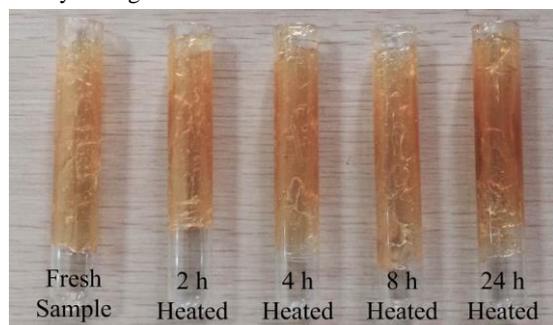


Fig. 2 Photographs of experimental samples

The color variation, which may be owed to the formation of oil separation or even oxidation products, is one of the most obvious signs of the variation of grease disperse system and component. Fig. 2 displays the optical photographs of fresh grease sample and heated grease samples with different test duration. It can be clearly seen that the color of the samples becomes slightly deeper along with the increase of heat treatment time. There is no significant variation of sample color after heat treatment for 0 h to 4 h, while the color displays slight variation after heat treatment for 8 h and 24 h. Previous studies^{17,18} have detected that the lubricating grease displays obvious thermal ageing phenomenon with the formation of thermal ageing products after heat treatment for long time, which needs to spend sufficient time (10 days¹⁷, 288 hours¹⁸). In this work, the heat treatment time is much less than the time of thermal ageing.

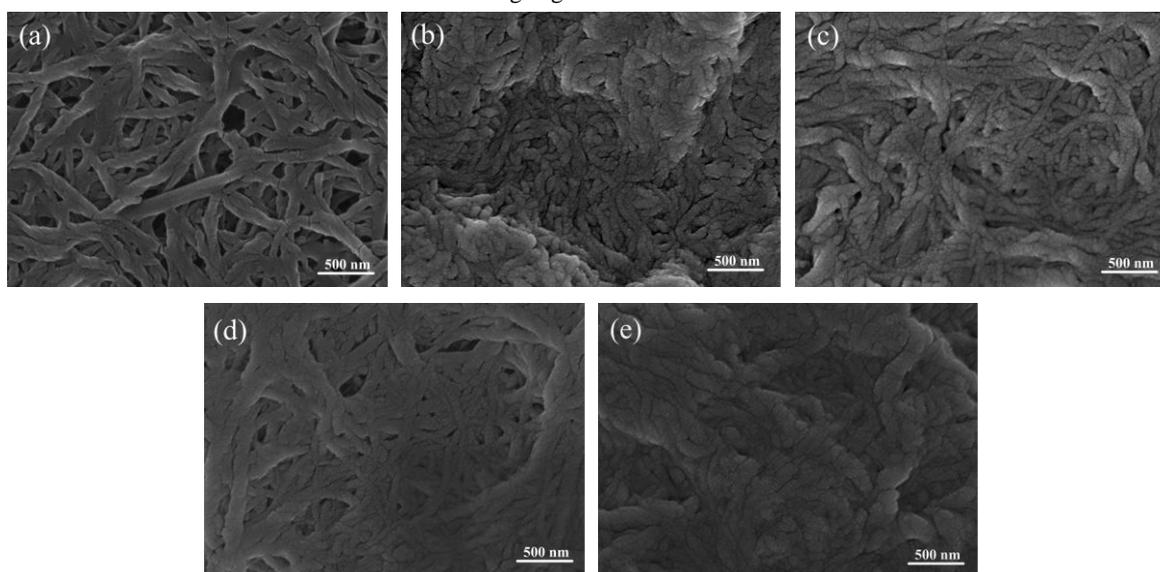


Fig. 3 Microstructure of all grease samples

(a) Fresh Sample; (b) 2 h Heated; (c) 4 h Heated; (d) 8 h Heated; (e) 24 h Heated

Figs.4a and 4b display the FTIR spectra of fresh grease sample and heated grease samples at 120 °C for 2 h, 4 h, 8 h and 24 h. It can be clearly seen that there are several intense peaks of all grease samples. The representative of characteristic peak is the specific functional group and bond, as described in Table 2.

As can be observed, the infrared spectra of heated grease

Therefore, the color variation may be attributed to oil separation of lubricating grease because the base oil displays a deeper color than fresh lubricating grease. When the oil separation appears and the base oil is unable to disperse again, the lubricating grease may display a deeper color. According to this extrapolation, it can be concluded that the oil separation increases along with the prolongation of heat treatment time for 0 h to 24 h. The results determine that the disperse system of lubricating grease has appeared visible variation, and the unique lubricating performance precisely connects with its disperse system. However, the accuracy of this extrapolation needs to be verified by experiments. Therefore, this variation should be investigated via the view of chemical and physical properties, and the lubricating performance could be evaluated by its tribological properties further.

Microstructure and FT-IR

Fig. 3 shows the microstructure of the experimental samples observed by FESEM, which were obtained at different heat treatment time. All experimental samples display similar microstructure with a high entanglement fibrous structure. It can be clearly seen that the microstructure of fresh grease sample displays very clear and high entanglement fibrous structure, as shown in Fig.3a. However, the entangled condition of 2 h to 24 h heated samples displays less agglomerated and lower degree. And the variation of fibrous structure becomes obvious with the increase of heat treatment time. The results explain that the physical entanglement of NLGI 3 lithium lubricating grease varied when the fresh samples undergo short time of heat treatment.

samples for 2 h to 24 h is basically in accordance with the infrared spectra of fresh grease sample, and no new intense characteristic peak appears in all range of wavenumbers.

Previous studies^{17,18} have detected that the new peaks at 1715 cm^{-1} and 1736 cm^{-1} , characteristic of C=O bond (ketones and esters, respectively) formed by oxidation of the sample,

will appear for the aged grease samples when the grease sample has the thermal ageing phenomenon. Meanwhile, the peaks of 1560 cm^{-1} and 1579 cm^{-1} , characteristic of the COO bond came from composition of thickener, will become weakened. Moreover, the peak of 1377 cm^{-1} , characteristic of a C-H bond came from the CH_2 group of base oil, will be strengthened due to the breakage of long molecular chain.

As can be observed in Figs.4a and 4b, the new peaks at 1715 cm^{-1} and 1736 cm^{-1} do not arise in the FTIR spectra. Therefore, the aged products (ketones and esters) would not be released from grease samples after heat treatment for 2 h to 24 h.

The results of FTIR indicate that thermal degradation of the grease samples has not appeared after heat treatment at $120\text{ }^\circ\text{C}$ for 2 h to 24 h. In other words, no obvious chemical structure variation appears at above operation condition. However, according to the results of microstructure, it can be noticed that highly entangled and agglomerated fibres are apparent for fresh grease sample, whilst lower density of fibres and less agglomerated are apparent after heat treatment. Therefore, the color of the samples becomes deeper slightly along with the increase of heat treatment time, which is attributed to a higher oil separation degree of the grease samples due to the variation of physical entanglement.

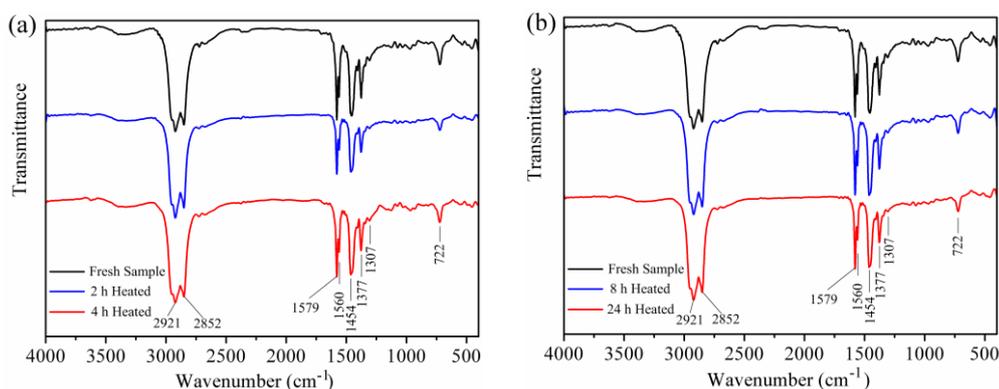


Fig. 4 FTIR spectra of all grease samples

Table 2 Characteristic peak and its corresponding molecular group and bond of grease samples FTIR spectra

Characteristic peak/ cm^{-1}	Functional group assignment	Band	Source
2921	Asymmetrical stretching vibration of CH_2 group	C-H	
2852	Symmetrical stretching vibration of CH_2 group	C-H	
1579	Asymmetrical stretching vibration of COO group	COO	Thickener
1560	Symmetrical stretching vibration of COO group	COO	Thickener
1454	Asymmetrical deformation vibration of CH_3 group	C-H	Base oil
1377	Bending vibrations of CH_2 groups	C-H	Base oil
1307	Twisting vibration of CH_2 group	C-H	
722	Overlapping of the CH_2 rocking vibration and the out-of-plane vibration of cis-disubstituted olefins	C-H	

Rheological properties

The rheological properties are major factors that determine the strength of the three-dimensional network of lubricating greases. Therefore, their rheological properties usually can be used to evaluate the role of lubricating greases in the lubrication of moving parts¹⁹. The storage (G') and loss (G'') modulus on frequency sweep test within the linear viscoelasticity range are used to estimate the strength of network structure. And the viscosity of constant shear rate can be used to simulate variation of viscosity in the lubrication process of stable revolve moving parts where the lubricating grease approximatively suffers constant shear.

In addition, the lubricating grease owns excellent viscosity-temperature characteristics, and detailed discussion has been carried out in many references^{5,6}. In other words, the

rheological property factors are not unique for a given system but a function of temperature. In order to evaluate the effect of heat treatment time on rheological properties, the experimental temperature was selected at $25\text{ }^\circ\text{C}$.

Fig. 5 shows the variation of the storage (G') and loss (G'') modulus on frequency within the linear viscoelasticity range for all grease samples as a function of heat treatment time. It can be observed that the values of the storage modulus (G') are always higher than those of the loss modulus (G'') in the frequency range between 10^{-1} s^{-1} and 10^2 s^{-1} . In addition, the values of both storage modulus (G') and loss (G'') modulus decreases with the increase of heat treatment time. Those results reveal the idea that both stored energy in elastic deformation process and lost energy in viscous flow process decreases when the fresh grease samples undergo short time of heat treatment. In other words, the fluidity of lubricating

grease is improved after heat treatment. This is mainly because the physical entanglements of lithium lubricating grease became less agglomerated and at lower degree after heat treatment.

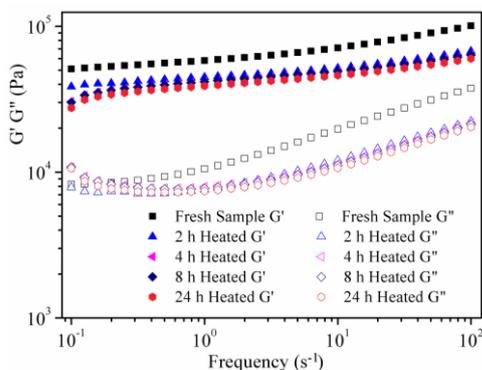


Fig. 5 Frequency sweep of all grease samples

Combined with the study of Mart n-Alfonso et al.¹⁹, the plateau modulus (G_N^0) is usually considered as a measure of the aggregation number among the dispersed structural units. This characteristic parameter is related to the strength of the microstructural network. Therefore, the plateau modulus (G_N^0) can be used as a reference parameter to evaluate the variation of viscoelastic behavior of experimental samples as a function of heat treatment times. Moreover, the plateau modulus (G_N^0) can be easily obtained through a straightforward method from frequency sweep data, as shown in Eq. (2).

$$G_N^0 = [G]_{\tan \delta \rightarrow \text{minimum}} \quad (2)$$

As can be observed in Fig. 6a, the plateau modulus (G_N^0) of fresh grease sample at 25 °C is obtained according to Eq. (2). Similarly, the plateau modulus (G_N^0) of other grease samples is given in Fig. 6b. It can be found that the values of plateau modulus (G_N^0) show a decrease tendency overall. And the data could be divided into three parts: obviously decreased (0 h-4 h), stable value (4 h-8 h) and slightly decreased (8 h-24 h). The results indicate that the entanglement strength of the grease microstructural network is evidently weakened after heat treatment for short time.

Fig. 7 displays the variation of viscosity of all grease samples at constant shear rate as a function of shear time. It can be clearly seen that the viscosity of grease samples decreases with the increase of heat treatment time. The results support the idea further that the fluidity of lubricating grease samples is obviously improved after heat treatment for short time.

Combined with the results of both the FESEM and FTIR analysis, the variations of plateau modulus (G_N^0) and viscosity are caused by the variation of physical entanglements rather than products of chemical variation. In other words, short time heat treatment can merely vary the physical entanglements and is not enough to alter chemical structure.

However, the specific lubrication properties of lubricating grease are generally related to the physical entanglements closely.²⁰ Therefore, the effect of heat treatment on lubricating properties of lubricating grease via the variation of physical entanglements should be studied by its tribological properties.

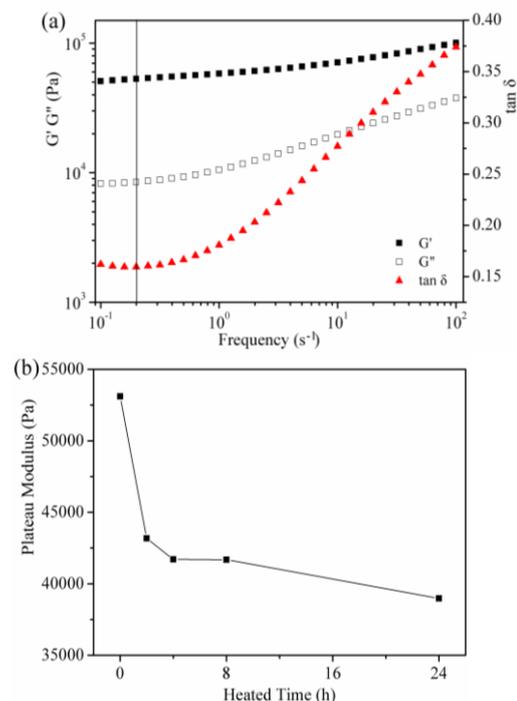


Fig. 6 The variation of plateau modulus as a function of heated time

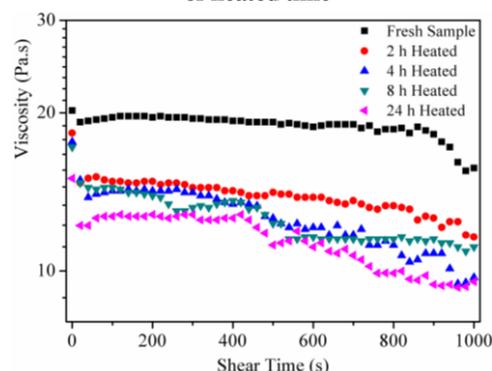


Fig. 7 The variation of viscosity for all grease samples (at constant shear of 100 s⁻¹)

Tribological properties

Fig. 8 shows friction coefficient of all grease samples as a function of friction time. It can be seen that the variation of friction coefficient could be divided two parts according to heat treatment time. From 0 h to 4 h, there is no obvious regularity of the friction coefficient. However, the samples after heat treatment for 8 h and 24 h exhibit lower friction coefficient with the increase of heat treatment time.

Fig. 9a and 9b display the WSD and the stable friction coefficient of all grease samples as a function of heat treatment time. It can be seen that the variations of both WSD and friction coefficient display well correspondence. In other words, as the friction coefficient decreases with the increase of heat treatment time, the value of WSD decreases. This character is mainly attributed to the unchanged component of all experimental samples, as the detailed result has been carried out in section 3.2.

Fig. 10 shows the morphologies of worn surfaces lubricated by different grease samples. It can be clearly seen that the worn surfaces of steel ball lubricated by different grease samples are very similar. There are no obvious differences among wear scars under different lubrication conditions. It can be concluded that all samples display the similar mechanism of antifriction and antiwear properties. When the friction coefficient decreases with the increase of heat treatment time, the lubrication condition of the rubbing surfaces could be improved. And the value of WSD will decrease. Therefore, the antifriction and antiwear properties of lithium lubricating grease are improved with the increase of heat treatment time.

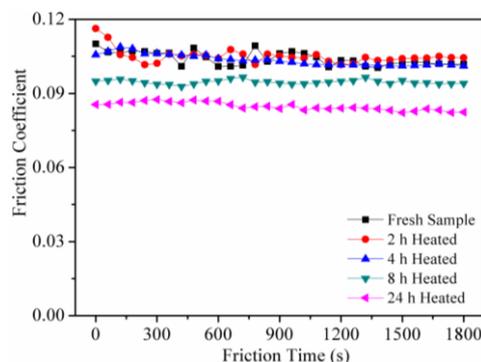


Fig. 8 Friction coefficient of all experimental samples (four-ball, 1450 r/min, 300 N, 30 min)

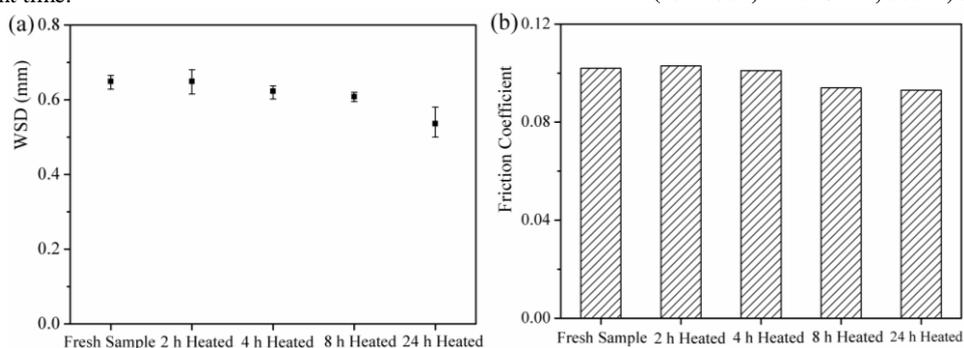


Fig. 9 WSD of all experimental samples (four-ball, 1450 r/min, 300 N, 30 min)

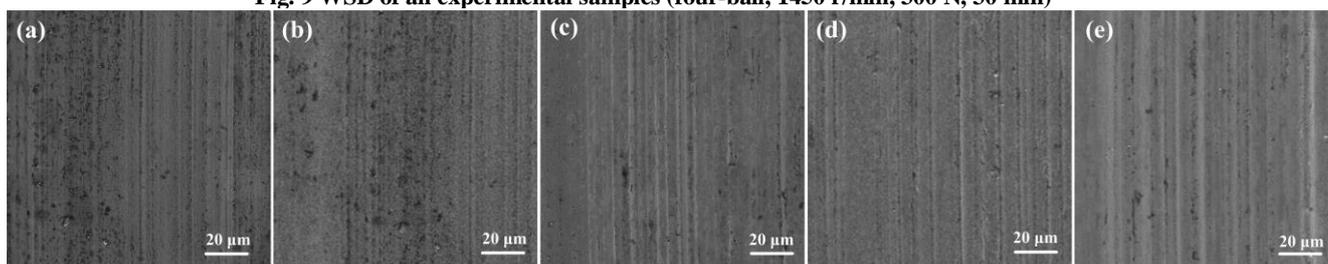


Fig. 10 Morphologies of worn surfaces lubricated by different grease samples

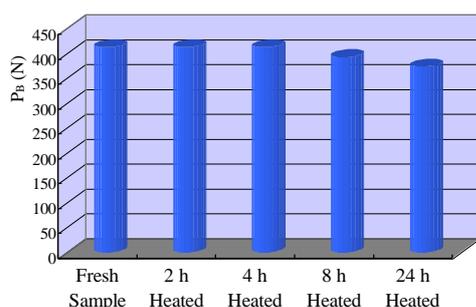


Fig. 11 P_B of all lithium grease samples (following the GB 12583-1998 standard)

Fig. 11 shows the extreme pressure load (P_B) of all grease samples. As a comparison, the P_B value of the fresh lithium lubricating grease is also given. It can be seen that the value of P_B keeps a stable value after heat treatment for 0 h to 4 h and declines slightly with the increase of heat treatment time from 4 h to 24 h. The results explain that the extreme pressure property, which is the load-carrying capacity of lubricating film, will be weakened when the lithium lubricating grease undergoes heat treatment, and it simultaneously decreases

with the increase of heat treatment time.

Lubrication mechanism analysis and discussion

The results of tribological properties display that the antifriction and antiwear properties have been improved with the increase of heat treatment time while the extreme pressure property gradually decreases. Meanwhile, the FESEM and FTIR results show that the chemical composition has not been changed during heat treatment, while the strength of grease fibrous structure displays decrease tendency. In other words, the lubrication performances of lithium lubricating grease vary by short time heat treatment, while it is not caused by the variation of its chemical composition but by its physical entanglement strength.

Several researchers studied that some polar groups (alcohols, acids and monoesters) could significantly improve the lubrication performance of oil lubrication by formation of stable boundary adsorption films on the rubbing surfaces.^{12-14, 21, 22} In this paper, the above results have excluded the possibility of polar group effect. According to the result of FTIR spectra, there is no obvious variation of all heated

samples. Especially, there is no new functional group appeared on the basis of the characteristic peak analysis. Therefore, the variation of lubricating properties is mainly caused by the variation of physical entanglement.

The replenished mechanism of grease lubrication is entirely different from that of oil lubrication. For oil lubrication, the lubricant of rubbing surface could be replenished easily with oil for its better fluidity. However, lubricating greases manifest weak fluidity, especially NLGI 2, 3 lubricating greases. Lubricating grease is easily lost from rubbing surface (the track of rolling bearing) either due to fluid displacement in the inlet region or as squeeze flow from the contact.²³⁻²⁵ The lubricant replenishment mechanism of the contact zone during grease lubrication was given by Cann²⁶. The lubrication process of grease in the track of rolling bearing could be divided into three stages: initial stage, layer formation stage and stabilized stage. In the initial stage, the grease undergoes continuous shear from inlet to contact zone. The majority of grease is rapidly pushed away from the track of rolling bearing. After this stage, only a fraction of grease deposits in the track and forms a grease layer. The structure of grease is broken down and free oil is squeezed from the contact with continued over rolling. In the stabilized stage, the grease of the side of the contact zone gradually releases base oil due to its structure broken down. The base oil or grease reflows into the contact to replenish the lubricant of rubbing surface. Therefore, efficient replenishment of grease lubrication plays an important role in keeping long time favorable lubrication of rubbing surface.

The results of friction and wear experiments showed that the antifriction and antiwear properties have been improved with the increase of heat treatment time. This mainly attributes to the minor non-chemical variation of grease during heat treatment for a short time. The mass loss rate and color contrast experimental results display that the lithium grease samples progressively lose weight and release base oil, which indicates that the lithium grease samples vary during heat treatment. The structural strength of soap fiber is weakened after heat treatment, and this has been confirmed by rheological property experiments. Besides, the viscosity of lithium lubricating grease at constant shear rate decreases with the increase of heat treatment time, and the grease displays better fluidity on the side of the contact. The base oil is more easily released from soap fiber of grease. Comprehensive above factors, the lubricant of rubbing surface could more easily be replenished with oil or grease reflow from the side of the contact in friction and wear experiments, which could improve the lubrication performance of heat treatment grease by formation of a stable replenishment circulation on the rubbing surfaces.

Combining the above results, it can be found that the lubricating properties of NLGI 3 lithium lubricating grease has not varied after 0-4 h heat treatment. And the grease displays a tiny non-chemical variation after 8 h and 24 h heat

treatment. Therefore, the heat treatment time should be controlled within 4 h or less and at 120 °C to ensure the impervious lubricating property of NLGI 3 lithium lubricating grease when the heat treatment is used to improve its pumpability.

Conclusions

According to the above results and discussions, the following conclusions can be obtained:

(1) The mass loss rate and color contrast analysis showed that the lithium lubricating grease samples progressively lose weight and release base oil. Visible variation of the lithium lubricating grease samples appear during heat treatment at 120 °C for 2 h to 24h, especially from 8 h to 24 h.

(2) The FTIR spectra of all heated samples have not changed relatively to fresh grease sample. Especially, there is no new functional group appeared on the basis of the characteristic peak analysis. However, the high entanglement network microstructure became less agglomerated and at lower degree after heat treatment for short time. And the strength of fibrous structure decreases with the increase of heat treatment time.

(3) The oil separation is facilitated and the viscosity at constant shear rate decreases after heat treatment at 120 °C for 2 h to 24 h. These performances realize efficient replenishment of base oil or grease for contact zone. The antifriction and antiwear properties have been improved by the formation of better oil reflow on the contact zone, which are unobvious during 0 h to 4 h. However, the extreme pressure property gradually decreases with the increase of heat treatment time.

(4) The heat treatment time should be controlled within 4 h or less and at 120 °C to ensure the impervious lubrication property of NLGI 3 lithium lubricating grease when the enhancing temperature technique is used to improve its pumpability.

Acknowledgments

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