Direct Observation of Thermo-Reversible Gel-Lubricants in EHL by FT-IR Micro-Spectroscopy

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The tribological properties of a new unique thermo-reversible Gel-Lubricant (TR Gel-Lube) were investigated. TR Gel-Lube which includes 10-40% of amide type gel agent in base fluid, is repeatedly able to change from gel-state to liquid-state at the melting point of its gel agent. TR Gel-Lube can be used as an impregnant for porous-material due to its physical state change. TR Gel-Lube indicated lower frictional and better lubricating properties than that of conventional greases. In particular, when TR Gel-Lube was sintered in a copper-type sintered bearing, a lower friction coefficient compared with oils was observed. In this study, to clarify the mechanisms of TR Gel-Lube, the behavior of gel agent on the EHL region was investigated by ball-on-disk tribometer. It was found that gel agent played a key role in the lubricating properties. Some mechanisms such as adsorbed film formation, smooth surface formation and solid like film formation are proposed.

Keywords: tribology, Gel-Lube, FT-IR, direct observation, low friction coefficient, sintered bearing

1. Introduction

Recently, functions of mechanical systems have been evolving, and today's lubricants are required to have high performance characteristics: long life, energy saving and anti-wear ability. On the other hand, conventional greases have some issues, such as oil separation and bad lubrication caused by thickeners.

To solve these problems, a new unique lubricant, which is called Thermo-Reversible Gel-Lubricant (TR Gel-Lube)^{1,2)}, was developed. TR Gel-Lube consists of base fluid and gel agent, and has some interesting characteristics. The most important characteristic is its reversible change of physical state, as shown in Fig.1. TR Gel-Lube can be used as an impregnant for porous-tribomaterial such as sintered bearings.

The aim of this study is to clarify the mechanisms of tribological properties of TR Gel-Lube. In previous work¹⁾, some basic lubricating and application test results using bearings were indicated comparing TR Gel-Lube with conventional greases. It was confirmed that TR Gel-Lube has better tribological properties than conventional greases. In particular, an extremely low friction coefficient resulting from TR Gel-Lube was obtained by sliding test using sintered bearings. These tribological properties are described in the next section before the experimental description.

To clarify the mechanism, the gel agent behavior on EHL was investigated by using interferometric and FT-IR techniques. Then some mechanisms were proposed in this study.

Fig.1 Physical state change of TR Gel-Lube by heating and cooling

2. TR Gel-Lube

2.1. Structure model of TR Gel-lube

TR Gel-Lube consists of base fluid and gel agent. Any base fluids, except high density fluorinated oil, can be used, such as mineral oil, PAO (Poly- α -olefin), ester oil, silicone oil and so on. The choice of gel agent is important because its molecular structure and melting point dominate the properties of TR Gel-Lube.

Fig. 2 shows a structure model of TR Gel-Lube. The gel agent has polar groups and lipophilic groups. TR Gel-Lube might be formed with base fluid in three-dimensional network of gel agent. It is thought that the network and gel state result from hydrogen bonds among the gel agents and van der Waals' force between base fluid and gel agents. Thermal stress and mechanical shearing affect these chemical bonds. The gel-state becomes liquid-state by heating up to the melting point of its gel agent. After thermal stress is removed, the gel-state is reformed again. The temperature of the physical state change depends on the molecule structure of the gel agent. In this study, aliphatic amide compounds were used as the gel agent.

Fig.2 Structure model of TR Gel-Lube

2.2. Characteristics of TR Gel-Lube

The characteristics of TR Gel-Lube are as follows; good tribological properties such as low friction coefficient, good lubricity, thick oil film formation, strong adhesive strength and long fatigue life^{1,2)}.

In our study, same samples consisting of common base oil were used. TR Gel-Lube consisting of 10% aliphatic-di-amide compounds (melting point:145°C) and poly- α -olefin base fluid (VG 68, including 1%mass of TCP) was prepared by heating up to the melting temperature. Li grease and urea grease with the same base fluid were used for comparison. These samples have the same consistency number (No. 2) as shown in Table 1. The same base fluid was used as a comparison in some tests.

Table 1 Properties of samples

	TR Gel-Lube	Li Grease	Urea Grease
Base fluid	90%	92%	88%
Di-amide	10%	—	—
Stearic Li	—	8%	—
Di-urea	—	—	12%
Consistency	268	279	284

Base fluid: PAO VG68 including 1% of TCP

Lubricating property was measured by SODA-type pendulum tester. The results of the pendulum friction test under boundary lubrication are shown in Fig. 3. The contact pressure was 0.97 GPa. The friction coefficient of TR Gel-Lube was 30% lower than that of the base fluid and conventional greases. The gel agent significantly decreased the friction resistance.

Fig.3 Friction coefficient by pendulum test

Sliding tests using two kinds of bearings were carried out in application^{1,2)}. The test conditions using thrust bearings are shown in Table 2. Small thrust bearings, inside diameter of $\phi 20$ mm, were used whereas the number of balls was decreased from 14 to 3. The specifications of the bearings are also summarized in Table 2. Before the test, 5 g of samples were packed in the bearings. Under these conditions, the bearings were continuously rotated for 168 hours. The failure rate, the percentage of breakdown during 168 hours, was determined.

Table 2 Test conditions using thrust bearing

Thrust bearing specifications	Type: 51104 thrust Balls: 14 (standard) → 3 Ball size: 5.556mm Inside:20mm, Outside:35mm
Load	4.4 kN
Rotation	1000 rpm
Temperature	Ambient
Sample	5g-packed
Test duration	168 hours
Evaluation during test time	No trouble → OK Trouble → NG
Failure rate	NG / Experimental

The results of fatigue life of the thrust bearings are shown in Fig. 4. It was found that TR Gel-Lube resulted in a low failure rate. On the other hand, the failure rate of urea grease was highest. It can be said that TR Gel-Lube indicated long fatigue life.

Fig.4 Results of fatigue life test of thrust bearing

The procedure using porous sintered bearings are as follows¹⁾. Copper alloy type sintered bearings were used in this study. TR Gel-Lube consisting of 40% of gel agent was penetrated about 20% volume of the bearing body under high temperature and vacuum conditions. The sliding test conditions and bearing specifications are shown in Table 3. In this test, the friction coefficient was continuously recorded.

Table 3 Test conditions using sintered bearing

Sintered bearing specifications	Type: Cu-Sn alloy Porous rate: 20vol% Diameter: inside 4.0 mm outside 7.0 mm Width 3.0 mm
Load	3.7kgf/cm ² (0.36 MPa)
Rotation	100 and 4000 rpm
Temperature	Ambient
Sample	Penetrated 20vol% of bearing body at 150°C under vacuum
Test duration	6 hours
Evaluation	Friction coefficient

The results of the sliding test using sintered bearings are shown in Fig. 5. At low sliding speed, the friction coefficient of TR Gel-Lube indicated 10^{-3} order value compared to 10^{-4} order of hydro-gel in the field of biotribology.

Fig.5 Friction coefficient of sintered bearing with some sliding speeds

3. Experimental

3.1. Apparatus

To clarify the tribological properties of TR Gel-Lube, the gel agent behavior on the EHL region was investigated using a ball-on-disk tribometer. Figs. 6 and 7 show the apparatus³⁾. A sapphire disk coated with chromium 100 mm in diameter and a steel ball 25.4 mm in diameter were used. They were rotated by separate motors independently. The chromium-coated sapphire disk was used for both the FT-IR micro-spectroscopy and optical interferometry because the decrease of FT-IR transmittance is only 2-3%. This experiment was carried under 0.13 m/s, 0.64 GPa. The tribometer was equipped with an FT-IR microscope. An infrared beam can be scanned along the lubricating direction as shown in Fig. 6. So main scanning was made from the center of the EHD contact to the inlet (-350 μ m position from center). The size of the infrared beam was fixed at 25 μ m $\times$ 25 μ m.

The test conditions are shown in Table 4.

Fig.6 Apparatus of tribometer equipped with FT-IR from side view

Fig. 7 Apparatus of tribometer equipped with FT-IR from top view

Table 4 Test conditions of EHL tribometer

Load	20 N
Rolling speed (FT-IR)	0.13 m/s
(Interferometry)	0.26 m/s
Slide/roll ratio	0%
Points of measurement distance from center of EHL	0, 50, 100, 150, 200 μm

3.2. Samples

In this work, same samples were used in Table 1. TR Gel-Lube, Li grease and Urea grease included common base oil and indicated the same consistency.

4. Result and discussion

4.1. Observation of gel agent behavior on EHL

Using FT-IR technique, the oil film formation and gel agent concentration on EHL were observed. Figs. 8 and 9 show the FT-IR spectrums of TR Gel-Lube in the center and inlet of EHL. The absorbed peaks due to N-H of gel agent and C-H of base fluid (PAO) were confirmed for both spectrums. It was observed that the gel agent was accurately introduced in the center of EHL.

Fig. 8 FT-IR spectrum of TR Gel-Lube under 0.13 m/s, 0.64 GPa at center of EHL

Fig. 9 FT-IR spectrum of TR Gel-Lube under 0.13 m/s, 0.64 GPa at 200 μm from center

The C-H peak area of TR Gel-Lube was thicker than that of general greases at all observed points as shown in Fig. 10. Especially, in the center of EHL, TR Gel-Lube was superior. Furthermore, the gel agent concentration of TR Gel-Lube, which is the calculated peak area ratio NH/CH, increased with approaching the center of EHL as shown in Fig. 11. These results indicate that in use of TR Gel-Lube, gel agents adsorb on metal surface at the center of EHL, so the oil film can be formed thickly.

Fig. 10 Changes in peak area against observed points

Fig. 11 N-H/C-H ratio change with measurement points on EHL

4.2. Film formation behavior on EHL

The long term film formation behavior on EHL was observed by interferometrical technique. The results of oil thickness change with passage of time are shown in Fig. 12. Initially, Li grease was worst at making the oil film. After 80 minutes passed, its oil thickness was thinner than that of base fluid. On the other hand, urea grease was the most stable. Its oil thickness was constantly thicker than those of the others. Gel-Lube, was very thick at the starting time. However finally, it was almost equal to base fluid. This phenomenon can be explained by the fact that TR Gel-Lube changed to soft gel state by shearing.

Fig.12 Change of oil thickness with passage of time

4.3. Mechanisms

TR Gel-Lube had good tribological properties compared with conventional greases. Based on observations of EHL behavior, the gel agents significantly play an important role in lubricating properties. The tribological properties of TR Gel-Lube can be explained by chemical and physical effects. The mechanisms of TR Gel-Lube, Li grease and urea grease are proposed as shown in Figs. 13, 14 and 15.

TR Gel-Lube includes a high percentage of gel agent. This gel agent can easily and strongly adsorb on metal surfaces with its own polar group. Also a stable film might be formed by network among the gel agent. It is believed that adsorbed film plays a role as an oiliness agent. This mechanism is chemical effect. From viewpoint of physical effect, it is believed that an ideally smooth surface is formed by TR Gel-Lube on rough metal surfaces. And it is reported that TR Gel-Lube becomes solid state under high pressure⁴⁾. Therefore, solid-like film might be formed. This physical effect protects during metal-to-metal contact. Additionally, in the case of sintered bearings, a drastically lower friction coefficient was observed. In this case, elastic deformation of porous body played an important role, as well as the characteristics of TR Gel-Lube. It is thought that this mechanism is similar to the biotribology of the human joint.

(a) Working as an oil agent

(b) Formation of smooth surface

(c) Formation of solid-like film

Fig.13 Mechanisms of TR Gel-Lube.

Fig.14 Mechanism of Li grease

Fig.15 Mechanism of urea grease

On the other hand, at first Li grease oil thickness was very thin and its friction coefficient was relatively high. It is thought that thickeners of Li grease are too large to be introduced into the EHL region, so oil starvation occurs. Also though urea grease oil thickness was the thickest, its friction coefficient was relatively high. It is assumed that thickeners can be introduced into the EHL region but they don't work for lubricity because of the urea structure.

5. Conclusions

The EHL behavior of a new unique lubricant, TR Gel-Lube was investigated using a basic tribometer. To clarify the mechanism, oil film formation on EHL was

observed by ball on disk tribometer. The following conclusions may be drawn from the results.

- The gel agent is accurately introduced in the center of EHL.
- The film thickness of samples are thin as follows:
Li grease < TR Gel-Lube < Urea grease
- Mechanisms of adsorbed film formation, smooth surface formation and solid like film formation are proposed.

6. References

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