Antiwear Property of Sericite-Added Grease

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The antiwear properties of sericite were compared with those of commercially available molybdenum disulfide and graphite by using a Soda-type 4-ball tester, when each solid lubricant was added to the lithium stearate grease. The oleophilically surface-treated sericite gave better antiwear properties and smoother worn surfaces than molybdenum disulfide and graphite did. This is due to the formation of a mixed lubricating film of the surface-treated sericite with soap micelles in the grease, because of its improved-affinity for the base grease. The surface-untreated sericite gave almost the same antiwear properties as molybdenum disulfide did.

1. Introduction

The good lubricities of solid lubricants such as molybdenum disulfide, graphite, and synthetic niobium sulfide, $Nb_{1+x}S_2$, are related to their layered crystal structures.^{1)~3)} In molybdenum disulfide crystal, there exist weak van der Waals forces between sulfur atoms in adjacent layers. This accounts for the easy shear and good lubricities of molybdenum disulfide.⁴⁾

Sericite also has a layered crystal structure like molybdenum disulfide. The layered crystal structure of sericite viewed along the *a*-axis has interlayer potassium ions balancing the negative layer charge. This ionic bonding between adjacent layers may suggest the easy slip by shear stress among thin and flat sericite particles rather than the shear between adjacent layers in sericite crystal. In the sericite/oil suspension systems, especially, the slip among sericite particles can be promoted by the prevention of particle coagulation due to the oleophilic surface treatment and by the existence of oil among the particles.⁵⁾

The suspension oils and greases of the surface-treated sericite (3–10 wt%) gave better antiwear properties than the base oil and base grease.⁵⁾ In the present study, the antiwear property of sericite was compared with those of commercially available molybdenum disulfide and graphite by a Soda-type 4-ball tester, when these solid lubricants were added to the lithium stearate grease.

2. Experimental

Sericite (average particle size, 0.3 μm , supplied by Nogami Kogyo Co., Ltd.) refined by elutriation

and flushing method, was surface-treated with oleylamine acetate to make it oleophilic. Commercially available graphite and molybdenum disulfide (average particle size, 0.7 μm) were used as reference solid lubricants.

The mixture (viscosity, 14.0 cSt at 40 °C, 3.65 cSt at 100 °C) of di-2-ethylhexyl sebacate (75 wt%) and mineral oil (25 wt%) was used as the base oil for the grease. The base grease was prepared from the mixture (80 wt%) and lithium stearate (20 wt%). The lubricants for the wear test were prepared by kneading of the base grease and 5 wt% solid lubricant.

Antiwear properties of the base grease and 5 wt% solid lubricant-added greases were examined by a Soda-type 4-ball tester, under the condition of oil pressure 1.5 kgf/cm² and 750 rpm for 30 min. All experiments were repeated three times and the measured values were taken an average. The wear scars of the balls were observed by using an optical microscope and a scanning electron microscope. The roughness curves of the worn surface were measured by the stylus method.

3. Results and Discussion

Table 1 shows the antiwear properties of the lithium stearate grease and greases containing 5 wt% solid lubricants. The surface-untreated

Table 1 Antiwear Properties of Solid Lubricant-Added Greases

Lubricants	Wear Scar Diameter, mm
Base Grease	0.77
5 wt% Surface-Treated Sericite	0.48
5 wt% Surface-Untreated Sericite	0.66
5 wt% Molybdenum Disulfide	0.68
5 wt% Graphite	0.93

Soda 4-Ball Test; 1.5 kgf/cm²; 750 rpm, 30 min

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Fig. 1 Micrographs of Wear Scars Produced in Base Grease and Solid Lubricant-Added Greases

sericite gave almost the same antiwear property as molybdenum disulfide did. The grease containing the surface-treated sericite gave less wear than the base grease and other solid lubricant-added greases. This is due to the lubricity of sericite itself, and due to its suspensibility in grease and the lubricity improved with the oleophilic surface-treatment.⁵⁾ The coagulation among the surface-treated sericite particles is unlikely because of their affinities for the base grease. The slip among the particles is promoted by the surface treatment that hinders particle-coagulation. Sericite, when it was added to a grease, exhibited, at least, the same, or better, antiwear properties than those of the commercial molybdenum disulfide and graphite applied here.

Micrographs and surface roughness profiles of the wear scars of the steel balls produced in the base grease and solid lubricant-added greases are shown in **Figs. 1** and **2**, respectively. The larger

worn surfaces with the base grease and graphite-added grease had metallic luster, and many grooves were formed on the whole worn surfaces. In this work, the effective antiwear properties were not obtained for graphite. The surface-treated sericite of all solid lubricants gave the smoothest surfaces as shown in **Figs. 1(b)** and **2(b)**. The worn surface was black when sericite particles were oriented on it. This suggests that sericite particles entered between the frictional surfaces, and thereby reduced wear and produced smoother surfaces.

The surface-untreated sericite and molybdenum disulfide gave the medium antiwear properties and surface roughnesses. Slightly worse antiwear properties of these solid lubricants, as compared with the surface-treated sericite, may be due to the difference in their suspensibilities in grease. This is suggested from the surface treatment effect of sericite.

Fig. 2 Surface Roughness Profiles of Wear Scars

Figure 3 shows scanning electron micrographs of the wear scars shown in Fig. 1, when the base oil of the grease was flowed out with dropping benzene-droplets onto the wear scars. In the base grease, fiber-like soap micelles were observed on the entire worn surface (Fig. 3(a)). The wear scar micrograph obtained with the grease containing the surface-treated sericite is quite different from those with other solid lubricant-added greases, as shown in Fig. 3(b). A mixed lubricating film of sericite particles with soap micelles may be formed on the frictional surfaces. The surface-treated sericite with oleylamine acetate has a great affinity for the base oil and lithium soap micelles and can form a stable and homogeneous suspension. The surface-treated sericite particles and soap micelles in this suspension adhered onto the frictional surfaces in the solid state, giving better antiwear properties.

Fig. 3 Scanning Electron Micrographs of Wear Scars Produced in Base Grease and Solid Lubricant-Added Greases (Magnification: $\times 10,000$)

For other solid lubricants, the worn surfaces dotted with solid lubricant particles were obtained as shown in Figs. 3(c)—3(e). These solid lubricant particles did not cover satisfactorily the whole frictional surfaces due to their particle coagulations and heterogeneous suspensions. The suspensibilities and lubricities of molybdenum disulfide and graphite may be improved with such surface treatment-effect as was obtained for sericite.

From the results described above, we conclude that: (1) sericite exhibits the same, or better, antiwear properties than those of commercial molybdenum disulfide and graphite; (2) the suspensibility of sericite in a grease becomes better with its improved-affinity for the base grease because of its oleophilic surface-treatment; and (3) the antiwear properties of sericite are improved due to the formation of a mixed lubricating film of the surface-treated sericite with the soap micelles in the grease. Good antiwear properties of sericite should be verified with additional experiments performed under various mechanical conditions.

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要 旨

セリサイト添加グリースの耐摩耗性

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親油化表面処理および未処理セリサイトがリチウムステアレートグリースに添加されたときの耐摩耗性が、曾田式四球試験機によって、市販の二硫化モリブデンおよびグラファイトの耐摩耗性と比較検討された。親油化セリサイトはベースグリースおよび他の固体潤滑剤と比べて、かなりの耐摩耗性を示した(**Table 1**)。未処理セリサイトは二硫化モリブデンと同程度の耐摩耗性であった。摩耗テストで得られた摩耗痕の顕微鏡観察(**Fig. 1**)によると、ベースグリースおよびグラファイト添加グリースによる摩耗面は金属光沢を呈し、摩耗面全体に条痕が生じていたのに対し、親油化セリサイト添加グリースによる摩耗面はセリサイト粒子の配向と思われるような黒色を呈していた。また未処理セリサイトおよび二硫化モリブデン添加グリー

スによる摩耗面状態はほぼ同等で、前二者の中間程度の状態であった。これらの摩耗痕に対応する表面あらさは**Fig. 2**に示されるが、**Table 1**および**Fig. 1**の結果と一致する。親油化セリサイトは他に比べて、よい耐摩耗性と非常になめらかな摩耗面を与えた。この理由として、摩耗痕中央部の走査型電子顕微鏡写真(**Fig. 3**)に示すように、親油化セリサイトは他の固体潤滑剤と比べてその表面がベースオイルおよび石けんミセルとの親和性が良いために、グリース中にはば一次粒子の状態に均一に分散し、しかも石けんミセルと混合潤滑膜を形成してよい耐摩耗性を与えたものと考えられる。これに対して他の固体潤滑剤は親和性に乏しいため一部凝集と分散状態が劣るために親油化セリサイトほどの耐摩耗性は得られなかった。

Keywords

Antiwear agent, Grease, Lubricant, Sericite, Solid lubricant, Tribology