Lubricities of Sericite-Suspended Oils and Greases

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High purity sericite having fine particle size, refined by flushing method and elutriation, was surface-treated to prepare its stable suspension in oil and grease. The lubricities of the sericite-suspended oils and greases were investigated by a pendulum type friction tester and Soda four-ball tester. With the sericite-suspended oils, fine and flat surface-treated sericite particles entered easily between frictional surfaces with oil flow, orienting nicely on them. The oriented sericite particles slipped over each other by shear stress to reduce friction and wear. In the sericite/grease system, the surface-treated sericite had an affinity for the soap micelles in grease, and formed a mixed lubricating film having high load-carrying capacity.

1. Introduction

The good lubricating properties of molybdenum disulfide and graphite are related to their layered crystal structures.1)-4) These solid lubricants have been added to lubricating oils and greases as additives, and used widely as solid films under conditions (e.g. space environments5)) where lubricating oils could not be applied. Recently, we have reported the lubricities of synthetic niobium sulfides with similar layered crystal structures.6),7)

Natural sericite also has a layered crystal structure. It is expected to be recognized as a solid lubricant, because of its thin and flat hexagonal platelike structure, and because it has better thermal stability as compared with those of molybdenum disulfide and graphite. However, the use of sericite as a solid lubricant has not been extensively investigated by the following reasons: (1) natural sericite includes comparatively large particles for a solid lubricant, (2) it contains impurities such as hard quartz and feldspar, and (3) stable dispersions or suspensions in oil are not obtainable because sericite has a poor affinity for oil.

In the present study, high-purity sericite of fine particle size (<2 µm) was obtained by elutriation and flushing method.8) The finely refined sericite was surface-treated with a surface active agent to facilitate its suspension in oil and grease, and the lubricities of these suspensions were investigated.

2. Experimental

2.1 Refining of Sericite

Sericite was supplied by Nogami Kogyo Co., Ltd. The refining of sericite formed in the zinc ore deposit of Tagokura mine was performed by a flushing method, to separate the very fine sulfide mineral particles of sphalerite and arsenopyrite from sericite.8) Large particles of sericite and other impurities were removed by elutriation. It was confirmed by electron microscopic observation that the refined sericite did not contain impurities. The chemical composition of the sericite obtained is shown in Table 1. The particle size of the sericite was below 2 µm in diameter (average particle size, 0.3 µm) (Fig. 1).

2.2 Preparation of Lubricants

Paraffinic oil (viscosity 20.1 cSt at 40 °C, 4.07

![Fig. 1 Electron Micrograph of Sericite](image)

| Table 1 Chemical Composition of Sericite |
|----------------------------------------|---|
| SiO₂                                  | 47.45 |
| Al₂O₃                                 | 31.19 |
| Fe₂O₃                                 | 2.32 |
| TiO₂                                  | 0.22 |
| CaO                                   | 0.08 |
| MgO                                   | 1.22 |
| K₂O                                   | 10.23 |
| Na₂O                                  | 1.18 |
| Ig. Loss                              | 4.82 |
| -H₂O                                  | 0.45 |

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cSt at 100°C, viscosity index (VI) 100) and di-2-ethylhexyl sebacate (11.6 cSt at 40°C, 3.2 cSt at 100°C, VI 149) were used as the base oils for the sericite-suspended oils and greases, respectively. n-Hexane was used as the vehicle of the suspension for the preparation of the sericite dry film.

The surface of sericite was treated with oleylamine acetate in order to make it oleophilic. The required quantity (3--30wt%) of the surface-treated sericite was kneaded in the n-hexane solution of 5.0wt% decaglyceryl decaoxlate (NIKKOL Decaglyn 10--0, HLB=12), and its mixture with 10 parts of di-2-ethylhexyl sebacate was kneaded until n-hexane evaporated. Finally, the sericite suspension was prepared by diluting the mixture with 90 parts of paraffinic oil. The sericite/n-hexane suspension was similarly prepared using n-hexane and methyl isobutyl ketone in place of paraffinic oil and di-2-ethylhexyl sebacate, respectively. The sericite-suspended greases were prepared by kneading sericite and lithium stearate (12wt%)/di-2-ethylhexyl sebacate grease (dropping point, 178, JIS K 2220), using a three-roll mill.

2.3 Lubricities

Extreme pressure test, on a Soda-type four-ball tester with bearing-steel balls of 1.91 cm (3/4 inch) in diameter was conducted at 200 rpm (sliding speed 12.7 cm/s). Tests were carried on until a scuffing or an abrupt increase in friction occurred, while increasing the load at a rate of 0.5 kgf/cm²/min (Table 2). The wear tests were conducted under conditions of 1.5 kgf/cm² and 750 rpm for 30 min. The wear scars on the steel balls produced in the wear tests were observed with an optical microscope and a scanning electron microscope.

The friction coefficients of the sericite-suspended oil and the sericite dry film were measured using a pendulum type friction tester. The sericite/n-hexane suspension was fed on the balls and roller pin, and the sericite dry film formed after the evaporation of n-hexane.

2.4 Oxidation Stability

Oxidation characteristics of sericite and molybdenum disulfide were examined by differential thermal analysis (heating rate, 5°C/min, in air).

<table>
<thead>
<tr>
<th>Table 2 Test Equipment and Conditions</th>
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<tr>
<td>Friction Coefficient</td>
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<tr>
<td>Wear (W. S. D.) (cm)</td>
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<tr>
<td>Load-Carrying Capacity (kgf/cm²)</td>
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</tbody>
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<tr>
<th>Table 3 Friction Properties of Sericite</th>
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<tr>
<td>Lubricant</td>
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<tr>
<td>Base oil</td>
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<tr>
<td>Sericite-suspended oil (3 wt%)</td>
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<tr>
<td>Sericite dry film</td>
</tr>
</tbody>
</table>

3. Results and Discussion

The friction properties of 3wt% sericite-suspended oil and sericite dry film are shown in Table 3. Sericite performed to lower the friction coefficient of the base oil, while the sericite dry film gave a still lower friction. These low frictions may be due to the layered crystal structure of sericite or the slip among the flat surface-treated sericite particles by shear stress.

Figure 2 shows the antiwear properties of the sericite-suspended paraffinic oils. At all concentrations, the sericite-suspended oils gave less wear as compared with that of the base oil. Wear, however, began to increase when the concentration of sericite exceeded 10wt%. The best antiwear properties were obtained in the range of 3 to 13wt%.

Micrographs of the wear scars of the steel balls produced in the base oil and sericite-suspended oils are shown in Fig. 3. The larger wear scar
surface produced in the base oil had a metallic luster, and many relatively large grooves were formed over the whole wear scar surface as shown in Fig. 3(a). The wear scar surfaces with the sericite-suspended oils were much smoother as compared with that produced in the base oil, and appeared black as sericite particles had adhered on the surfaces (Figs. 3(b)—3(f)). This suggests that sericite particles entered between the frictional surfaces and thereby reduced wear.

Typical scanning electron micrographs of the wear scar surfaces with the sericite-suspended oils are shown in Figs. 4—6. The trace of oil flow and
adhesion of the sericite particles were observed well near the oil inlet zone and on the wear scar surface and its circumference (Fig. 4). Flat sericite particles oriented nicely in piles on the frictional surface (Figs. 5 and 6). The size and shape of the sericite particles on the wear scar surface (Fig. 6) is very similar to those of the original particles (Fig. 1). The surface-treated sericite particles were probably supplied to the frictional surfaces in their original fine particle form because particle coagulation is unlikely due to their affinity for the base oil. These facts may suggest that sericite particles, oriented on the frictional surfaces, slip over each other to result in low friction and wear. The slip among the surface-treated sericite particles is surely apt to occur as compared with that among the ionic bonded layers in crystal caused by shear stress. This slip among particles could be promoted by the prevention of particle coagulation due to the surface treatment and the existence of oil among them.

**Table 4** Effect of Surface Treatment of Sericite on Its Lubricity

<table>
<thead>
<tr>
<th>Lubricants</th>
<th>Load-Carrying Capacity a, kgf/cm²</th>
<th>Wear Scar Diameter b, mm</th>
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<tbody>
<tr>
<td>Base grease</td>
<td>3.0</td>
<td>0.66</td>
</tr>
<tr>
<td>Surface-treated sericite (13 wt%)</td>
<td>8.5</td>
<td>0.33</td>
</tr>
<tr>
<td>Surface-untreated sericite (13 wt%)</td>
<td>7.5</td>
<td>0.56</td>
</tr>
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</table>

*a*: 200 rpm, 0.5 kgf/cm²/min  
*b*: 750 rpm, 1.5 kgf/cm², 30 min

The effect of the surface treatment of sericite on lubricities was examined when the treated sericite was added to lithium stearate/di-2-ethylhexyl sebacate grease (Table 4). The grease containing the surface-treated sericite gave better load-carrying capacities and antiwear properties. The surface treatment of sericite may give a more stable and homogeneous suspension. The surface-treated sericite particles are apt to be supplied uniformly to the frictional surfaces because of their affinity for the base oil and soap micelles. This is supported by the results obtained in the sericite-suspended oil system.

**Figures 8 and 9** show the effect of addition of the surface-treated sericite on the lubricities of lithium stearate/di-2-ethylhexyl sebacate grease. The sericite-added greases showed improved antiwear properties in a higher range of concentrations as compared with that of the sericite-suspended oils, as shown in Fig. 8. However, the wear obtained in the sericite/grease system was much higher than that with the sericite-suspended oils at all concentrations. On the contrary, much better load-carrying capacities were obtained with the sericite-suspended greases as shown in Fig. 9. These facts may be attributed to the differences in the ease of supplying the sericite to frictional surfaces and the state of the lubricating films at high pressures. In the sericite/oil system, sericite
particles can easily enter between frictional surfaces with oil flow and orient on the surfaces as shown in Fig. 6. The lubricities of the sericite-suspended oils, therefore, may be mainly due to the slip among sericite particles. In the sericite/grease system, the surface-treated sericite particles have an affinity for soap micelles in grease, and form their mixed lubricating film as shown in Fig. 10. It is considered that at higher loads this mixed film transforms to the solid state to give higher load-carrying capacities.

Oxidation stabilities of sericite and molybdenum disulfide were examined by differential thermal analysis (Fig. 11). With molybdenum disulfide, an exothermic peak due to oxidation began to appear at about 375°C. Sericite showed no exothermic peak even at 620°C, indicating higher oxidation stability. It is expected that sericite would be a good solid lubricant for high temperature applications.

4. Conclusion

From the facts described above, we conclude that, (1) stable and homogeneous sericite-suspended oils and greases can be obtained provided that adequate surface treatment of sericite is applied and a suitable surface active agent is selected as a dispersant, (2) in the sericite/oil system, sericite particles, oriented on frictional surfaces, slip over each other by shear stress, giving good lubricities, (3) in the sericite/grease system, the surface-treated sericite particles and soap micelles form a mixed lubricating film which give high load-carrying capacities, and (4) sericite has better oxidation stability as compared with that of molybdenum disulfide.

Acknowledgement

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the electron microscopic observation of the wear scar surfaces.

References
4) Tsuya, Y., ASLE Trans., 15, 225 (1972).

Keywords
Additive, Lubricant, Lubrication, Sericite, Sericite-suspended oil, Tribology