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Dynamics of Solid Dispersions in Oil During the Lubrication of Point Contacts, Part II—Molybdenum Disulfide

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DYNAMICS OF SOLID DISPERSIONS IN OIL DURING THE LUBRICATION OF POINT CONTACTS, PART II - MOLYBDENUM DISULFIDE

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ABSTRACT

The dynamics of MoS_2 particles in a mineral oil dispersion are studied in the same manner as reported in Part I for graphite dispersions. A Hertzian contact consisting of a steel ball in contact with a glass disk is lubricated with MoS_2 dispersions and observed by optical microscopy at various slide/roll conditions. In general the behavior of MoS_2 and graphite are similar. That is, the solids tend to enter the contact and form a film on the contacting surfaces whenever a rolling component of motion is used, but solid particles seldom enter the contact during pure sliding. MoS_2 has more pronounced plastic flow behavior than graphite. However, the polished steel ball is more readily scratched by MoS_2 than by graphite. Under the conditions of these studies, lower friction and wear are observed with pure oil rather than with the dispersions. However under other conditions (such as different contact geometry or rougher surfaces) the solid lubricant dispersions might be beneficial.

INTRODUCTION

Molybdenum disulfide (MoS_2) in dry form, has the ability to form continuous, low shear strength, but adherent films between contacting metal surfaces in motion and thus provide excellent lubrication properties. Because of these properties the lubricating effectiveness of MoS_2, in dry form, has been intensively studied for many years. In more recent years studies have also been done on the lubrication characteristics of oils, especially mineral oils, with MoS_2 as an additive.

The conclusion drawn from most of these studies is that under boundary lubrication conditions, a reduction in wear and usually friction occurs with the addition of MoS_2 to mineral oils (1-9). These studies have shown that an MoS_2 film forms from the MoS_2 particles dispersed in the oil. This film reduces the metal-to-metal contact under boundary lubrication conditions. In addition, it has been found that the oil viscosity markedly influences the effectiveness of MoS_2 in oils. Usually MoS_2 has been found to be more effective in low viscosity oils than in high viscosity oils (10).

In reference 11 it has been shown that the beneficial effects of MoS_2 in pure mineral oils gradually increase with concentration. The percent improvement of the lubricating effectiveness of oils containing MoS_2, however, levels-off as the concentration of MoS_2 increases. The effects of particle size on lubricating effectiveness of MoS_2 dispersions has been investigated in reference 12. This investigation has shown that coarser

MoS_2 dispersions give higher wear values than MoS_2 dispersions of finer particles especially at relatively high loads. However, there are indications that, with rougher surfaces, coarser MoS_2 particles will show better wear performances than smaller particles.

The addition of MoS_2 to oils may not always have beneficial effects. Studies have shown, for example, that the addition of MoS_2 can have beneficial, neutral, or detrimental effects depending on the type and concentration of oil-soluble additives present in the oil (13-15). It is also probable that any good or bad influences of solid lubricant additives to oil depend upon factors such as contact geometry, surface topography, lubrication regime (boundary, mixed, EHD), slide/roll ratios, and operating conditions.

The scope of this study is restricted to observing MoS_2 dispersions in one type of contact geometry (the Hertzian contact of a polished steel ball on a glass flat) under conditions of pure rolling, a slide/roll ratio of one, and pure sliding. Very low surface velocities are employed to assure boundary lubrication conditions. The contact is observed by optical microscopy in a manner similar to that described in reference 16 for observing the dynamics of dry MoS_2 under pure sliding conditions.

EXPERIMENTAL APPARATUS

The basic components of the apparatus used consist of a bearing ball which rides against a Pyrex disk. The Hertzian contact was viewed through the Pyrex disk by means of a microscope. Photomicrographs of the contact were taken under various operating conditions.

The general configuration of the experimental apparatus is shown in figure 1. This apparatus was used in the companion study of graphite dispersions (17) and in EHD studies and is described in (18). The only modification made for the study of dispersions is the addition of a variable speed dc motor which was directly coupled to the Pyrex disk. With this modification the apparatus could be used in studies involving pure rolling, combined rolling and sliding, and pure sliding conditions. The pure sliding data were obtained by rotating the disk and keeping the ball fixed. All the data presented in this study were obtained at $25\pm2^\circ$ C and a relative humidity of 30 ± 5 percent.

TEST MATERIALS

The AISI 52100 bearing steel balls have a hardness of 65 R_c and an exceptionally smooth surface finish of 0.018 μm (0.7 μin.) rms. The disks are Pyrex glass with a surface finish of 0.03 μm (1.2 μin.) rms. A more detailed description of the specimens is given in reference 17.

The dispersions were made by using commercially available, lubricant grade MoS_2 powders and super-refined paraffinic mineral oils. Table 1 gives the particle sizes and typical analysis and chemical specifications of the MoS_2 powders while table 2 gives data for the two base oils. The three grades of MoS_2 powders and the two oils were used to make dispersions of 0.5, 1, and 3 percent (by weight).

PROCEDURE

The experimental procedure was the same as that described in reference 17, including the experimental sequence given in table 2 of that paper. As in that study, very low velocities were used so that the dynamics of the dispersed solids could be readily observed by optical microscopy and to minimize elastohydrodynamic lubrication. All experiments were therefore performed in the boundary lubrication regime.

RESULTS

Dynamics of MoS_2 Dispersions

Figures 2 to 6 show the distribution of MoS_2 dispersions in and around Hertzian contacts under various conditions. The inlet on all these figures is to the left of the contact. The original magnification in all photomicrographs of the Hertzian contacts was 150X. As indicated in table 1, average particle sizes of MoS_2 are 0.35 μm for suspension grade, 0.70 μm for medium grade, and less than 50 μm (by sieve analyses) for coarse grade powders. The largest particles of coarse MoS_2 actually observed with the microscope were about 40 μm.

Pure rolling. - Figure 2 shows contacts under pure rolling conditions subjected to a load of 2 kg and surface velocities of $u_1 = u_2 = 0.0021$ m/s. The MoS_2 concentration in figures 2(a) to (c) is 0.5 percent and in figures 2(d) and (e), it is 3 percent. Under these conditions of pure rolling at low velocities, the suspension grade MoS_2 particles are gradually packed between the two rolling surfaces and eventually a continuous film of MoS_2 is formed. Film continuity depends on the concentration of MoS_2 and particle size. Figure 2(c) shows that, with a relatively low concentration and large particles, it is more difficult to form a continuous MoS_2 film. At the higher concentration, the continuity of the film formed from coarse powder improves (fig. 2(e)). It was also observed that the MoS_2 films form less readily as the rolling velocity is increased. At higher rolling velocities the MoS_2 particles have less tendency to adhere to the contact areas and are more likely to be swept around the contact than through it. It should be noted that the separation of the rolling surface in figure 2 is primarily due to the MoS_2 film since a theoretical estimate of the nominal film thickness with the more viscous (150 cS) oil, for $u_1 = u_2 = 0.0021$ m/s and a load of 2 kg, gives a value of less than 0.0063 μm (0.25 μin.). This nominal film thickness is much less than the combined rms surface roughness of the surfaces in contact.

Combined slide/roll. - Figure 3 shows photomicrographs of the contact with the surfaces subjected to a combined rolling and sliding motion. The disk velocity is 0.0021 m/s while the pheripheral velocity of the ball is 0.0007 m/s to give a slide/roll ratio of 1. With the possible exception of figure 3(a), it is seen that an MoS_2 film separates the surfaces for all conditions considered in figure 3. By comparing figures 3(a) and (b) and figures 3(c) and (d) it can be concluded that, for an MoS_2 concentration of 0.5 percent, an MoS_2 film is more easily formed when the lower viscosity oil is used than when the higher viscosity oil is used. However, for an MoS_2 concentration of 3 percent the film formation is not visibly influenced by the viscosity of the carrier oil (compare figs. 3(e) and (f) and figs. 3(g) and (h)). Therefore, the viscosity of the oil does not seem to be as important when higher concentrations of MoS_2 are used. Complete coverage of the Hertzian contact with an MoS_2 film occurs even when coarse grade powders are used if the concentration is high enough. This can be seen by noting the relatively thick MoS_2 film and track shown in figure 3(i) for 3 percent coarse grade MoS_2. As with the pure rolling case, however, continuous films are difficult to form from the larger particles.

By increasing the velocity of the disk to 0.0063 m/s and that of the ball to 0.0021 m/s, the slide/roll ratio remains one but the dynamics of the MoS_2 dispersions are quite different. Figure 4 shows photomicrographs for such velocities. This figure shows that the track which existed at the lower velocities has almost vanished. By comparing figures 4(a) and (b) again it is seen that there is more MoS_2 in the contact when using the lower viscosity oil than when using the higher viscosity oil.

Pure sliding. - The distribution of MoS_2 during pure sliding at 0.0021 m/s and at a 1 kg load is shown in figure 5. It is noted from this figure that accumulation and packing of MoS_2 at the inlet occurs. The packing becomes increasingly apparent as the particle size of the dispersed powder and the load are increased (figs. 5(b) and (c)). Note that unlike the pure rolling or combined rolling and sliding cases, no continuous film of MoS_2 is formed in the contact. The MoS_2 particles tend to pack and coalesce at the inlet, and the inlet serves as a reservoir from which relatively small amounts of MoS_2 are drawn into the contact. However, most of the MoS_2 goes around the contact.

Figure 6 gives examples of the contacts at a higher sliding velocity of 0.0146 m/s. Figure 6(a) shows that, even with a small concentration of suspension grade powder in the lower viscosity oil, a considerable accumulation of MoS_2 occurs at the inlet but again very little enters the contact. In fact, there is evidence of back flow at the inlet which tends to carry particles to the edges of the inlet where they either accumulate or flow around the contact while the center of the inlet region is relatively devoid of MoS_2. Figure 6(b) for 3 percent coarse grade MoS_2 and a higher load of 4 kg also does not show much evidence of MoS_2 in the contact in spite of a considerable build-up at the inlet. Considerable wear is also evident which verifies the lack of lubricant in the contact.

Coefficient of Friction

As in the case of the graphite dispersions described in Part I, friction coefficients during pure rolling and also at a slide/roll ratio of one were not measurably influenced by adding up to 3 percent MoS_2 to the carrier oils. Friction coefficients during pure rolling were 0.002±0.001. For a slide/roll ratio of one, friction coefficients were 0.04±0.01. However, during pure sliding, measurable but inconsistent differences were recorded.

Representative data for the coefficient of friction as a function of load during pure sliding are shown in figure 7. These data were taken during each experiment in a test series after the coefficient of friction had reached a steady state condition. Again, the results were essentially the same as had been observed for graphite dispersion. That is: No clear beneficial or detrimental effect of the solid lubricant additive on friction was observed. Usually, the friction was lower with the carrier oil alone than with the dispersions. This may be a result of lubricant starvation of the contacts caused by the solid lubricant build up at the inlet. The wear observations indicate a similar trend. As expected for boundary lubricated contents, friction was generally lower at the higher sliding velocity.

Wear

Figures 8 and 9 show the wear scars on the tool steel balls used in the sliding experiments. Oblique illumination was used when the photomicrographs were taken. Therefore, the highly polished areas appear black while the wear marks or scratches, which scatter light into the objective lens of the microscope, appear as bright streaks on the surface of the ball. From these photomicrographs, the following observations are made: (a) comparison of figures 8(a) and 9(a) to the other photographs show that less surface damage occurred with the carrier oil alone than with the dispersions; (b) as might be expected, less wear occurs with the higher viscosity oil; (c) with suspension grade MoS_2, wear is about the same for 0.5 and 3.0 percent concentration - compare figures 8(b) and (d); (d) more wear occurs with coarse grade MoS_2 than with the finer grades - compare figures 8(c), 8(f), and 9(c) to the other figures.

The wear scar shown in figure 9(c) was sputtered with xenon and an Auger Electron Spectroscopy analysis was conducted. This analysis indicated that there is a thin film of MoS_2 on the surface of the wear scar. No indication of molybdenum or sulfur that would suggest the presence of MoS_2 was detected on the ball surface outside of the wear scar.

DISCUSSION

The results presented in this paper are mainly concerned with the dynamics of MoS_2 dispersions in and around a concentrated contact. No attempts have been made to conduct experiments over a long period of time or at higher rolling and/or sliding speeds to more completely simulate the dynamic conditions which exist in practice. As stated previously, with higher speeds, observations of dispersions would have been very difficult without sophisticated high-speed photography.

Generally, comments made in the Discussion of reference 17 about the dynamics of graphite dispersions also apply to the dynamics of MoS_2 dispersions. However, some differences do exist and they will be discussed below. With suspension grade particles and at low sliding speeds the MoS_2 particles tend to coalesce and pack in front of the inlet while graphite particles will accumulate at the inlet but will generally not coalesce and tend to have continuous individual motion in front of the inlet. Even though the amount of graphite and MoS_2 which entered the contact was relatively small, MoS_2 was more likely to form a partial or complete film in the contact than graphite. These observations probably result from the fact that MoS_2 particles tend to flow and to coalesce into coherent thin films more readily than graphite. Such differences in the dynamic characteristics of MoS_2 and graphite have also been observed when these solid lubricants are used in dry contacts (16).

It is not unusual for MoS_2 to give better wear protection than graphite in dry contacts. However, for the operating conditions and duration of the tests reported in this paper, MoS_2 dispersions tend to be more abrasive than graphite dispersions. The relative abrasiveness of MoS_2 and graphite in oil dispersions can be seen by comparing the wear scars presented in this paper to those presented for graphite dispersions in reference 17. However, the least wear is obtained with the base oil alone as the lubricant. Therefore, no short-term beneficial effects were derived by adding either of the solid lubricants to the base oil.

These results should not be over-generalized; other contact geometries, rougher initial surface finishes, or higher surface velocities might produce different results. For example, the influence of surface topography on the transport of MoS_2 particles into and around the contact has not been fully explored. It is plausible that with surfaces rougher than the highly-polished ones used in this study, more solid lubricant would be drawn into the contact, even under pure sliding conditions. A rougher surface on the moving specimen of the sliding combination, in particular, would perhaps mechanically force the particles into the contact.

CONCLUSIONS

The lubrication of the Hertzian contact of a polished steel ball in contact with the flat surface of a glass disk was observed by optical microscopy. The major observations were:

1. Under pure rolling conditions and relatively low speeds, the MoS_2 particles are gradually packed between the two rolling surfaces and eventually a track of MoS_2 is formed whose continuity mainly depends on the concentration and particle size of MoS_2 in the oils. With a relatively low concentration and large particle size it is more difficult to form a continuous MoS_2 film. The MoS_2 film is also more difficult to form as the rolling speed increases. The higher fluid flow rates and increased back flow at the inlet associated with higher rolling velocities are apparently disruptive to the formation of continuous solid films on the surface.

2. An MoS_2 film is also formed under combined rolling and sliding conditions and low speeds. This film is more easily formed when the lower viscosity base oil is used. Again MoS_2 films are more difficult to form as the speed is increased.

3. Under pure sliding conditions, no visible, continuous MoS_2 films are deposited on the surfaces of the contact. Instead, MoS_2 particles accumulate and coalesce at the inlet, or simply sweep around the outside of the circular contact area. The MoS_2 accumulation is most pronounced at low sliding velocities and when coarse MoS_2 particles are used. The accumulation at times actually blocks oil from the inlet and increases lubricant starvation of the contact.

4. Auger electron spectroscopy after sliding experiments indicates that some MoS_2 is present on the ball wear scar although no continuous film is visible by optical microscopy.

5. The coefficient of friction is generally lower at higher sliding speeds. In general it can also be stated that the coefficient of friction is lower when the base oil alone is used than when MoS_2 is added to the base oil.

6. More wear occurs with dispersions of coarse grade MoS_2 than with dispersion of either suspension or medium grade MoS_2. In addition, as the concentration of coarse grade MoS_2 increases, wear also tends to increase.

ACKNOWLEDGEMENT

The authors wish to express their appreciation to John Ferrante at NASA-Lewis for conducting an Auger Electron Spectroscopy analysis on the wear scars.

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TABLE 1. - COMPOSITIONS AND PARTICLE SIZES OF MoS_2 POWDERS

	Analysis (spec.) wt. percent		
	Suspension grade	Medium grade	Coarse grade
MoS_2	(97.5 min)	(98.0 min)	(98.2 min)
Acid insoluble	0.35 (0.50 max)	0.35 (0.50 max)	0.35 (0.50 max)
Iron	.15 (0.20 max)	.15 (0.20 max)	.15 (0.20 max)
Molybdenum trioxide	.05 (0.15 max)	.03 (0.05 max)	.01 (0.05 max)
Water	.05 (0.15 max)	.00 (0.05 max)	.00 (0.05 max)
Oil	.25 (0.40 max)	.25 (0.40 max)	.03 (0.05 max)
Carbon	1.20 (1.50 max)	1.20 (1.50 max)	1.00 (1.50 max)
	Particle sizes		
	Typical average (Fisher)-0.35 μm	Typical average (Fisher)-0.70 μm	Larger than 150 μm - 0 percent
			+75-150 μm - 5 percent
	Maximum (Fisher)-0.45 μm	Maximum (Fisher)-0.85 μm	+50-75 μm - 10 percent
		Minimum (Fisher)-0.55 μm	-50 μm - 85 percent

TABLE 2. - FLUID PROPERTIES OF BASE PARAFFINIC MINERAL OILS

Property	Typical fluid properties	
	Oil I	Oil II
Gravity, °API	30.9	30.2
Viscosity at 98.9° C, cS	9.44	15.0
Viscosity at 37.8° C, cS	78	150
Viscosity index	106	106
Pour point, °C	-12	-12
Flash point, °C	232	238
Fire point, °C	277	293
Sulfur, wt., percent	0.02	0.02

TABLE 3. - TEST SERIES

Experiment no.	Load, kg	Maximum Hertz stress, N/m^2	Speed, m/s		Slide/roll ratio $(u_1 - u_2)/[0.5(u_1 + u_2)]$	Comments	Duration of test, min.
			Disk u_1	Ball u_2			
1	2	$5.0x10^8$	0.0021	0.0021	0	Pure rolling	8
2	2	$5.0x10^8$	.0021	.0007	1	Rolling and sliding	8
3	2	$5.0x10^8$	.0063	.0021	1	Rolling and sliding	5
4	1	$4.0x10^8$	.0021	0	2	Pure sliding	8
5	1	$4.0x10^8$	.0063				5
6	1	$4.0x10^8$	.0146				5
7	2	$5.0x10^8$	.0021				8
8	2	$5.0x10^8$	.0063				5
9	2	$5.0x10^8$	.0146				5
10	4	$6.3x10^8$	.0021				8
11	4	$6.3x10^8$	.0063				5
12	4	$6.3x10^8$	.0146				5

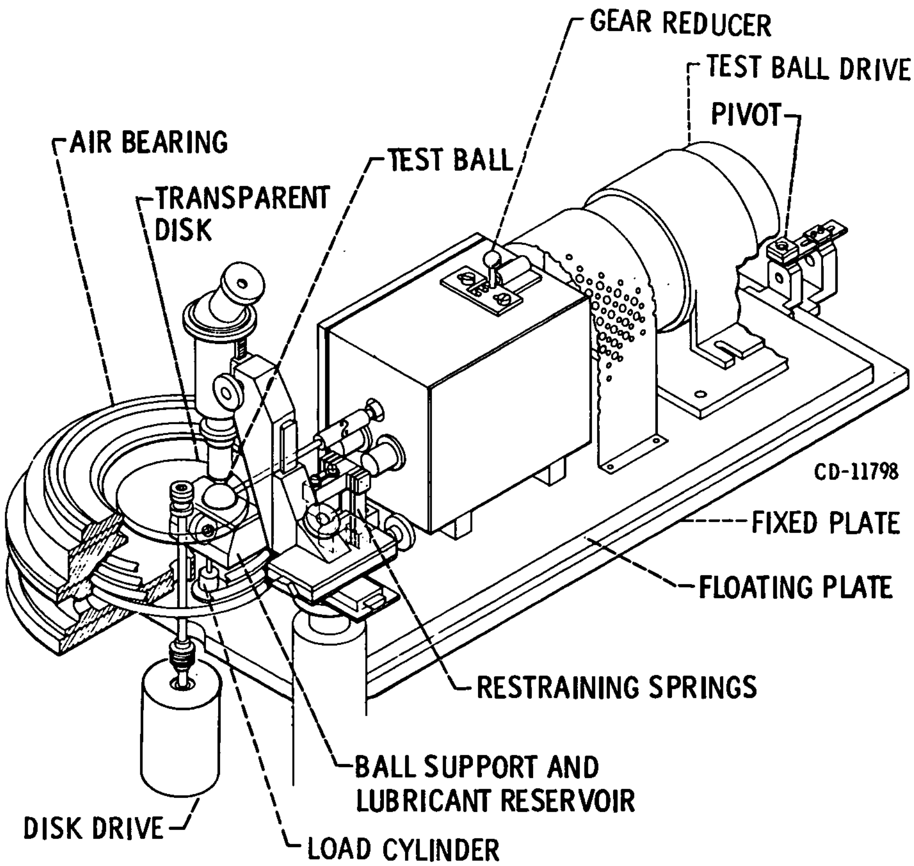
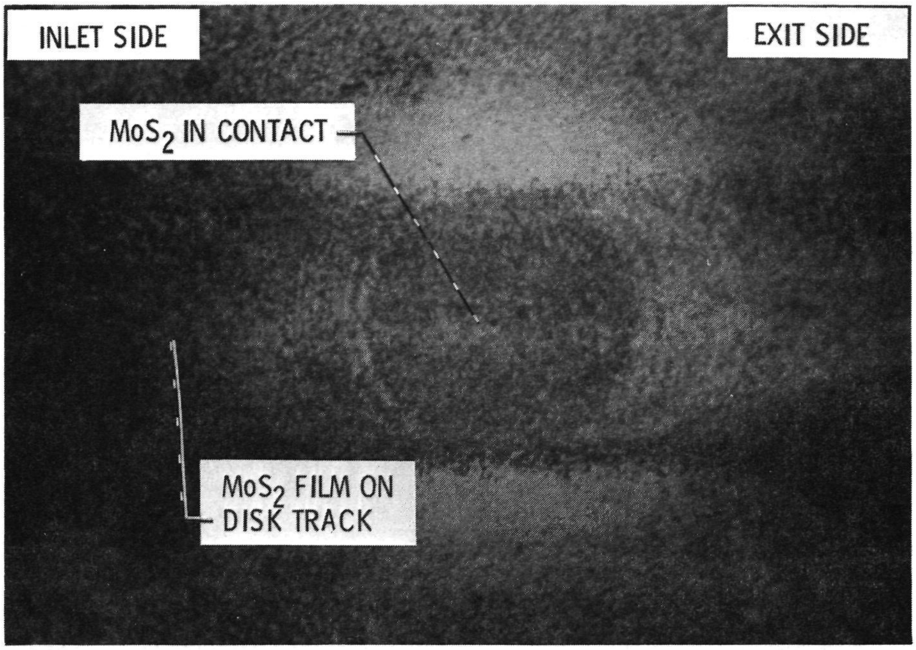
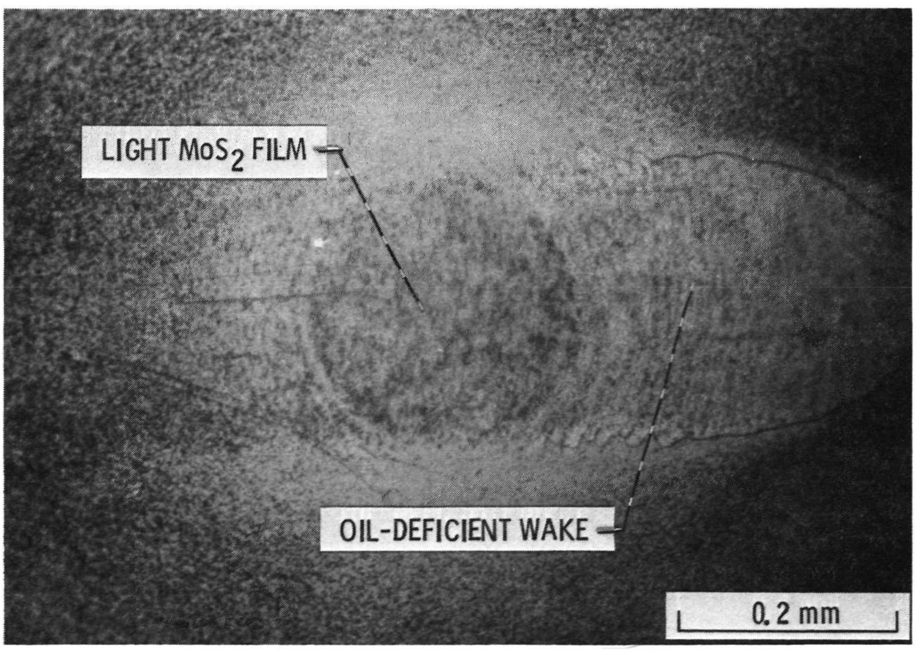


Figure 1. - Optical EHD rig.

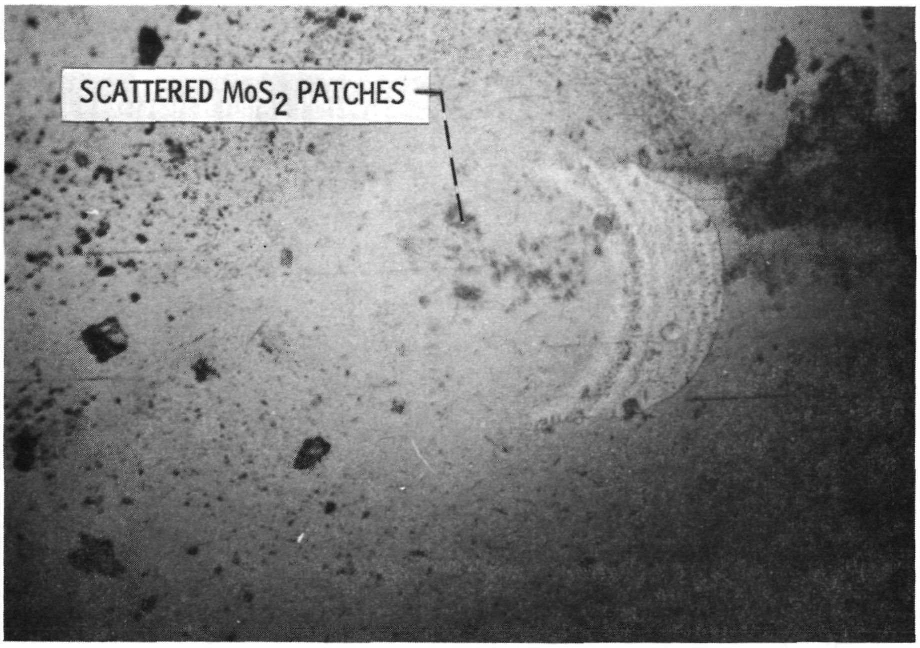


(a) 0.5% SUSPENSION GRADE IN 78 cS OIL.

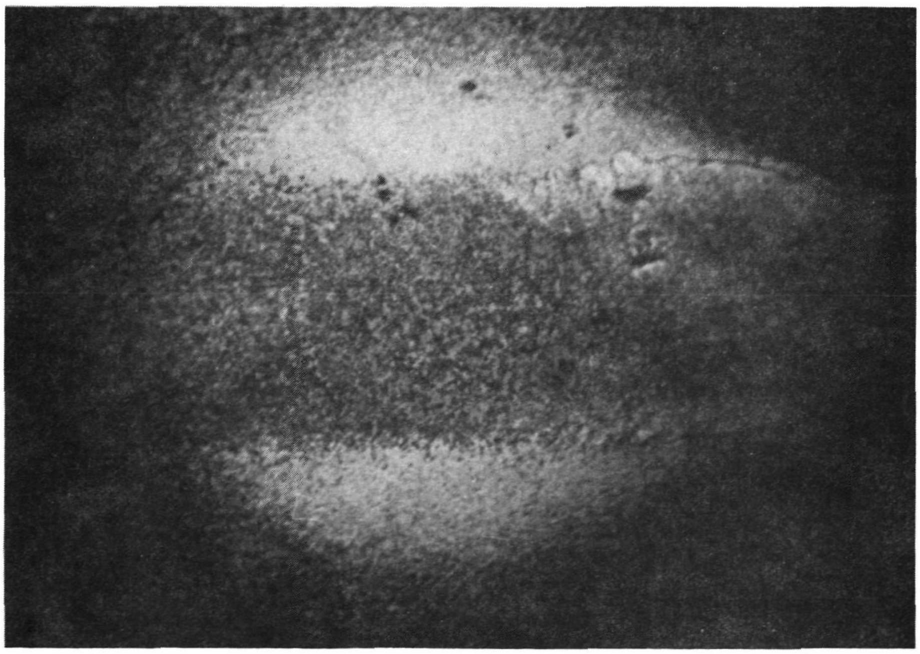


(b) 0.5% SUSPENSION GRADE IN 150 cS OIL.

Figure 2. - MoS_2 distribution during pure rolling as viewed at 150X original magnification 0.0021 m/s entrainment velocity, 2 kg load.

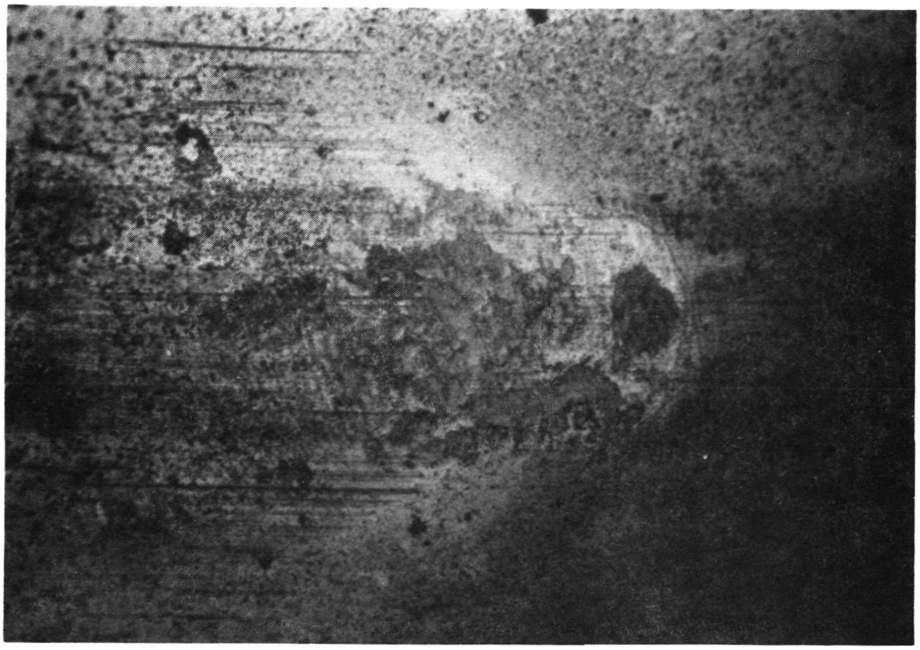


(c) 0.5% COARSE GRADE IN 150 cS OIL.



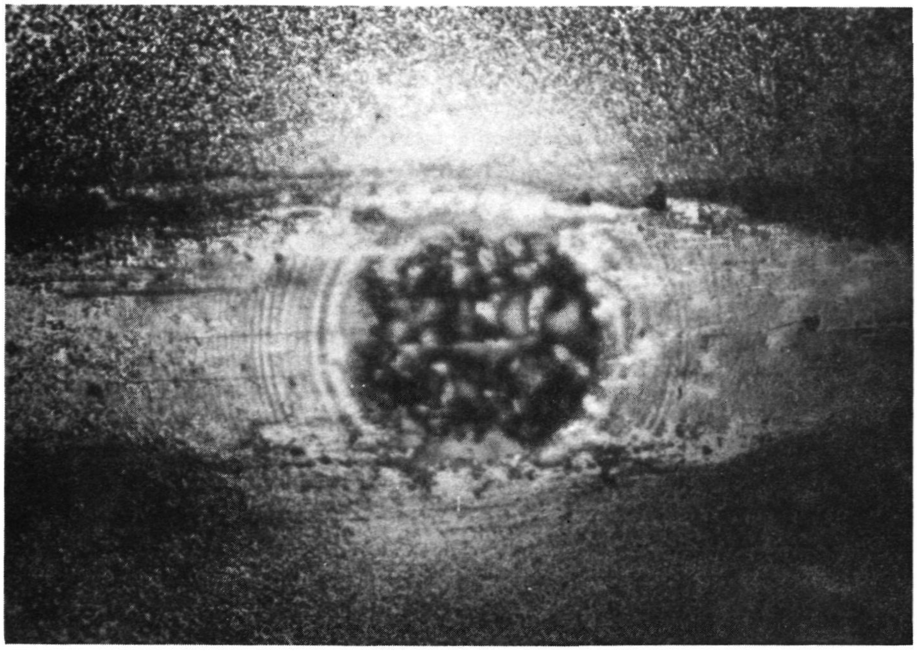
(d) 3% SUSPENSION GRADE IN 150 cS OIL.

Figure 2. - Continued.

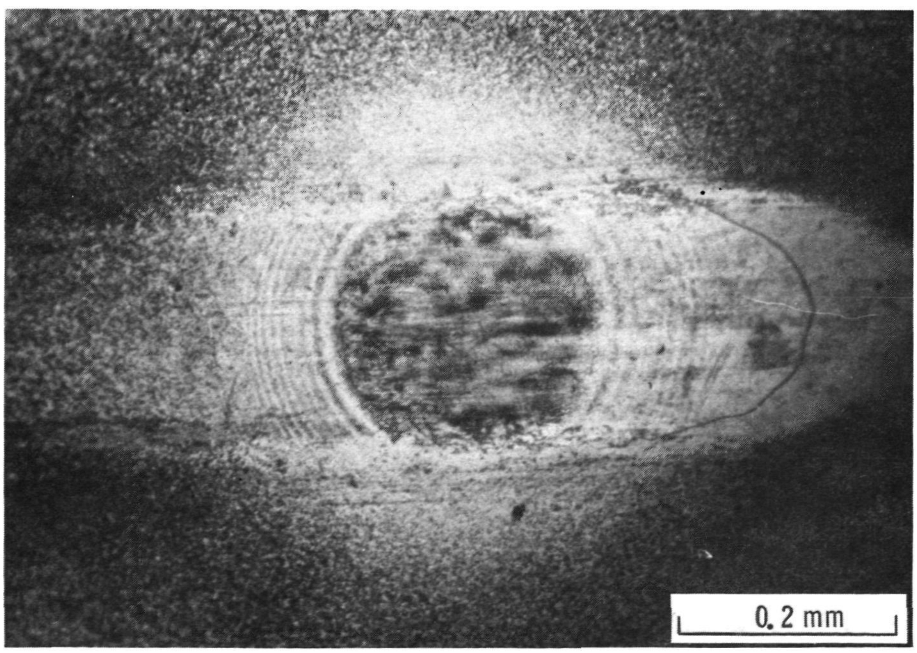


(e) 3% COARSE GRADE IN 150 cS OIL.

Figure 2. - Concluded.

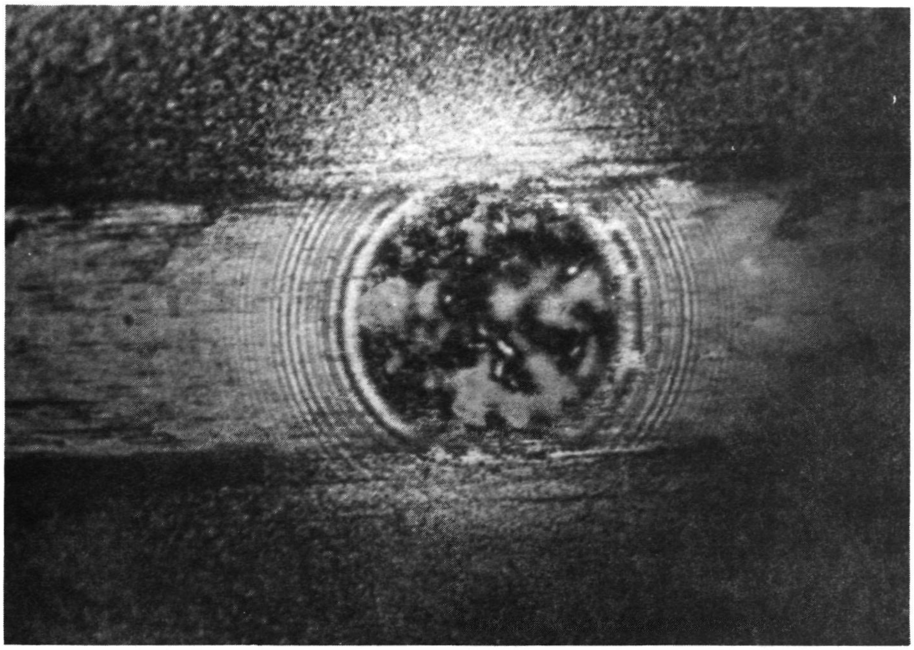


(a) 0.5% SUSPENSION GRADE IN 78 cS OIL.

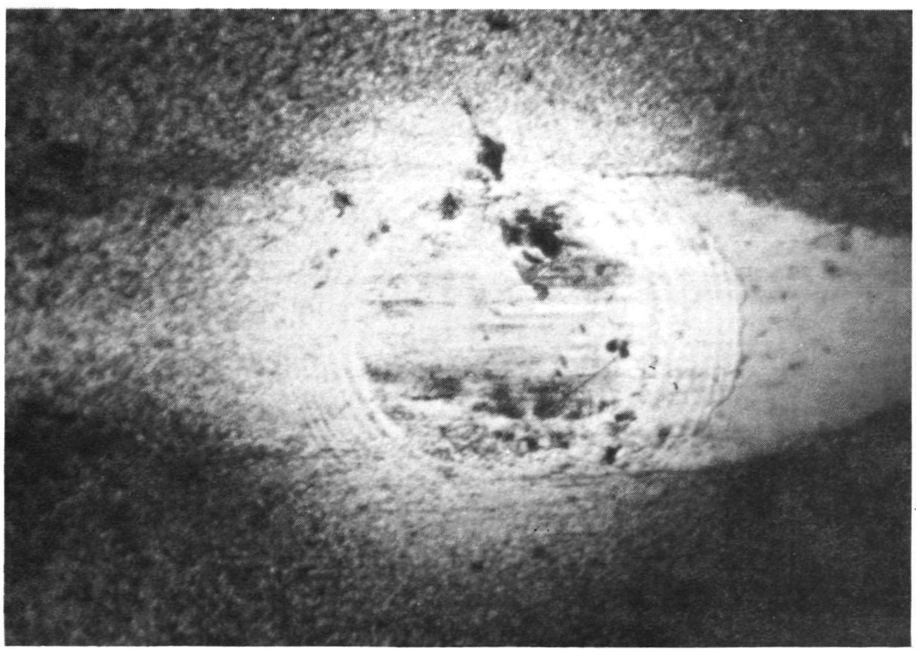


(b) 0.5% SUSPENSION GRADE IN 150 cS OIL.

Figure 3. - MoS_2 distribution at a slide/roll ratio, $\Sigma = 1$. $U_1 = 0.0021$ m/s, $U_2 = 0.0007$ m/s, entrainment velocity $\bar{U} = 0.0014$ m/s, 2 kg load.

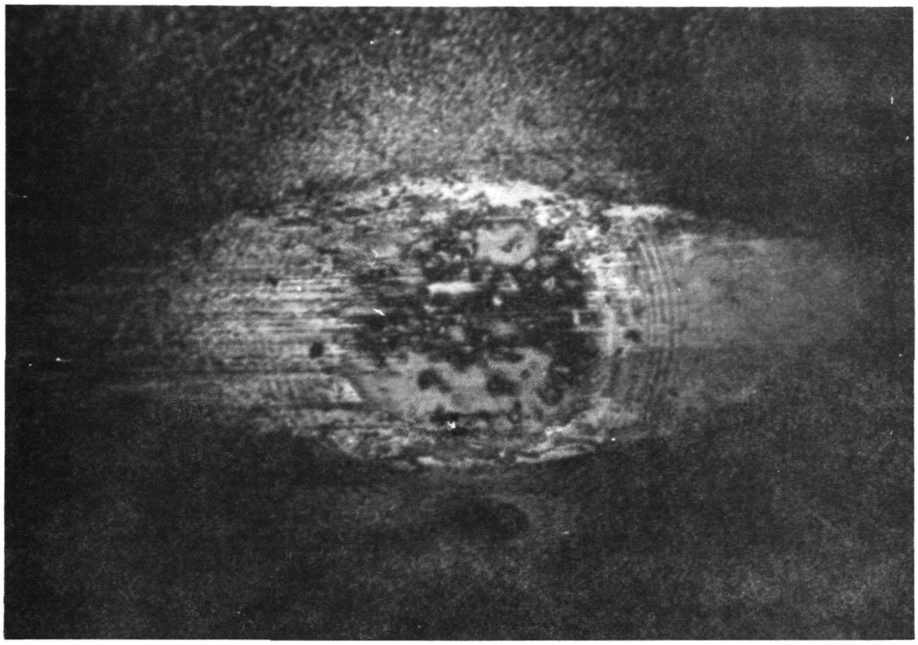


(c) 0.5% MEDIUM GRADE IN 78 cS OIL.

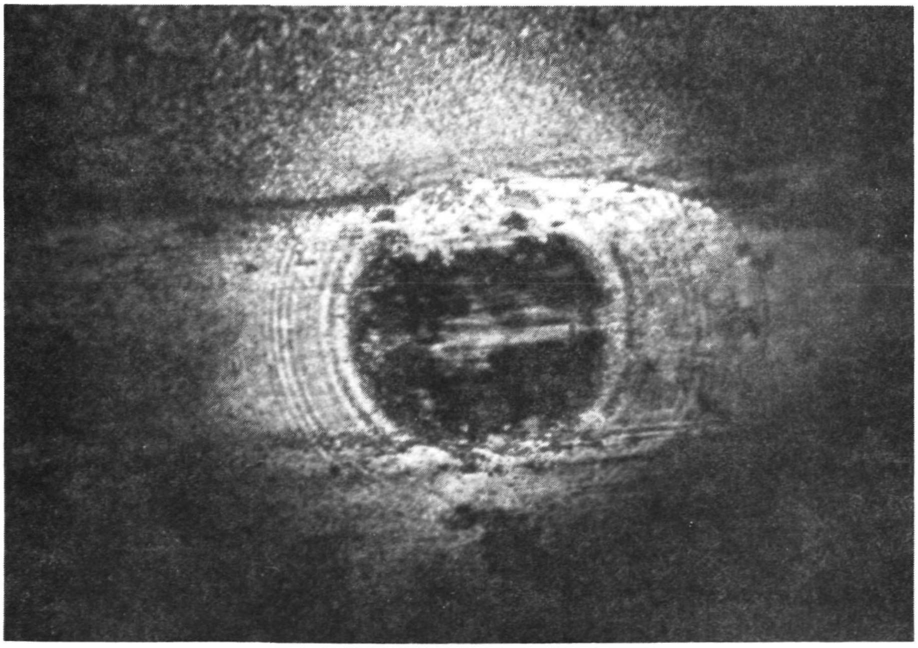


(d) 0.5% MEDIUM GRADE IN 150 cS OIL.

Figure 3. - Continued.

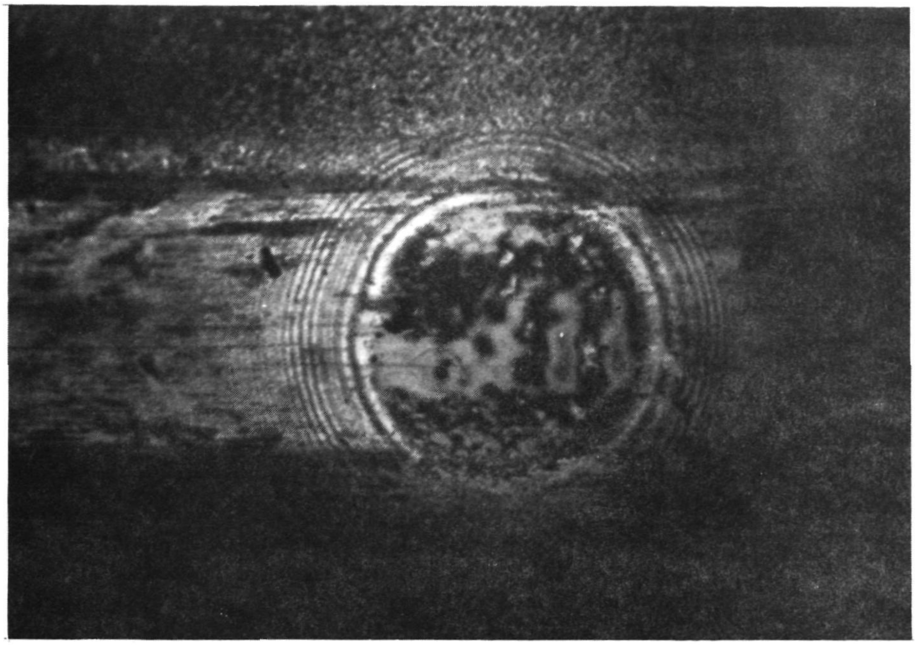


(e) 3% SUSPENSION GRADE IN 78 cS OIL.

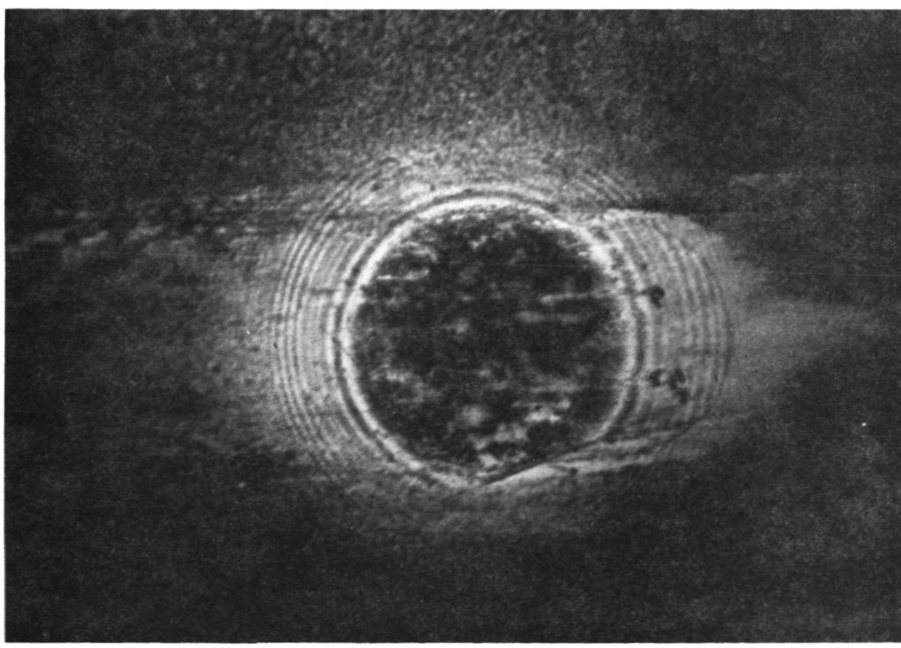


(f) 3% SUSPENSION GRADE IN 150 cS OIL.

Figure 3. - Continued.

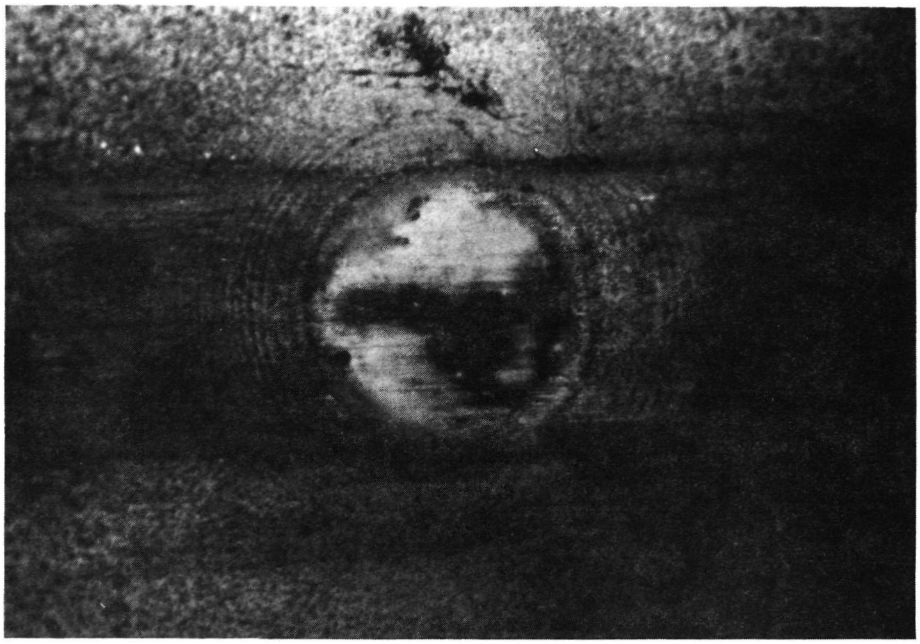


(g) 3% MEDIUM GRADE IN 78 cS OIL.



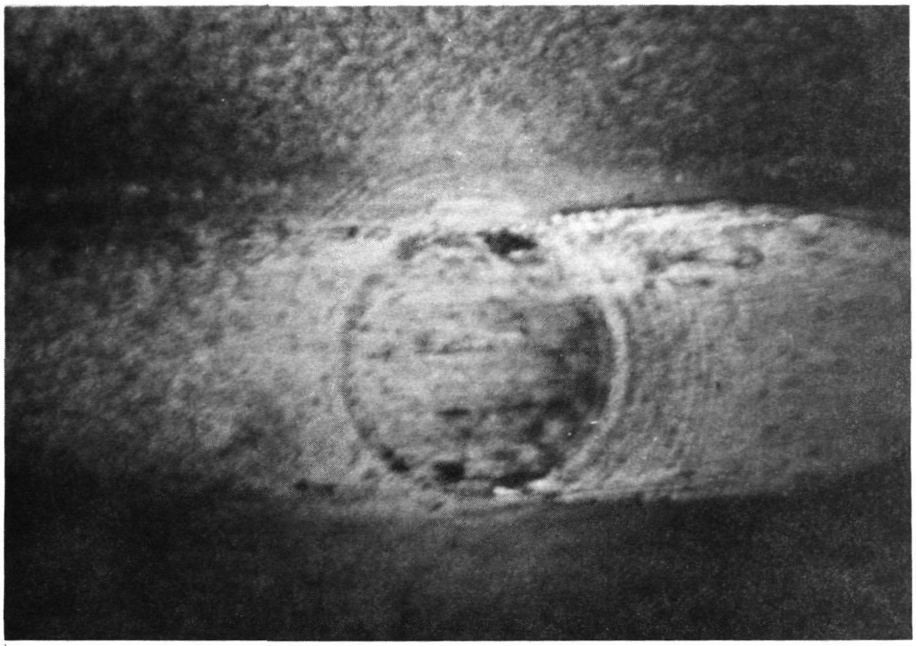
(h) 3% MEDIUM GRADE IN 150 cS OIL.

Figure 3. - Continued.

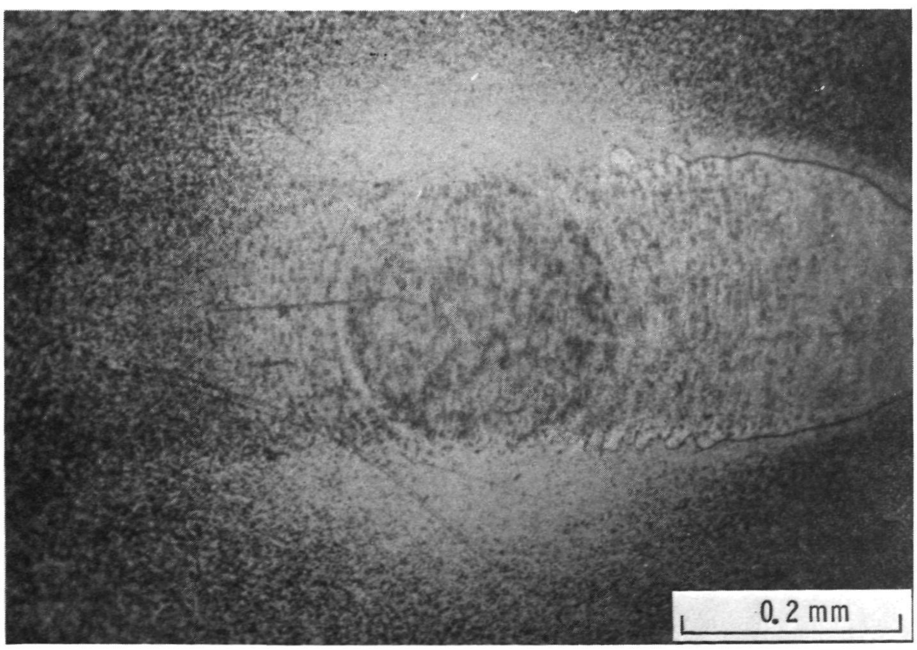


(i) 3% COARSE GRADE IN 78 cS OIL.

Figure 3. - Concluded.

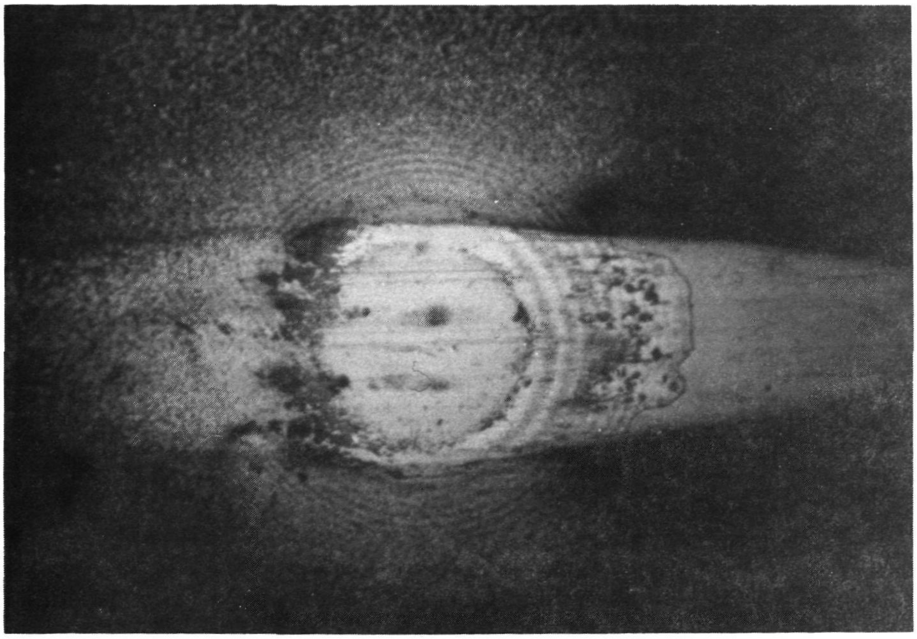


(a) 0.5% SUSPENSION GRADE IN 78 cS OIL.

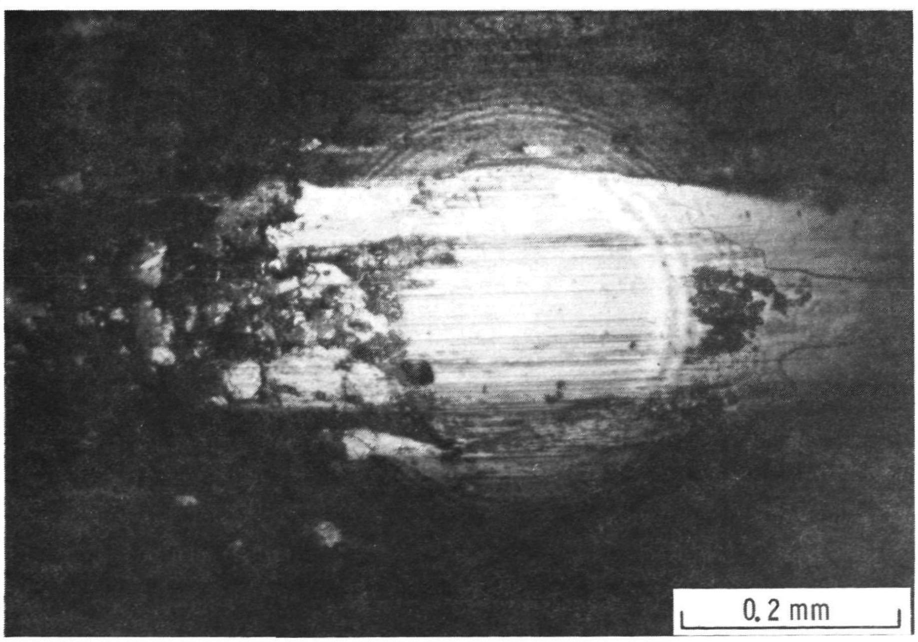


(b) 0.5% SUSPENSION GRADE IN 150 cS OIL.

Figure 4. - MoS_2 distribution at a slide/roll ratio, $\Sigma = 1$. $U_1 = 0.0063$ m/s, $U_2 = 0.0021$ m/s, entrainment velocity $\bar{U} = 0.0042$ m/s, 2 kg load.

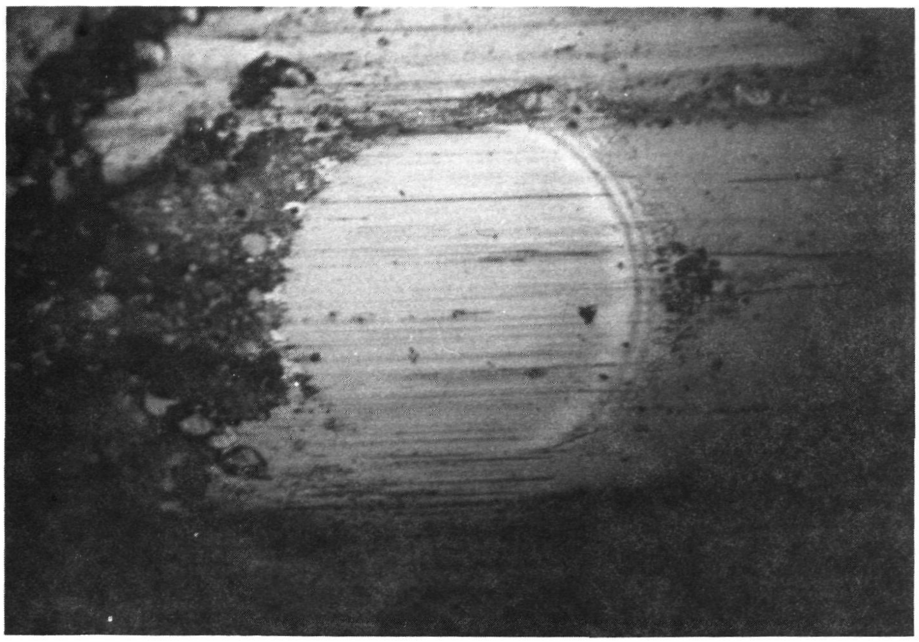


(a) 0.5% SUSPENSION GRADE IN 150 cS OIL, 1 kg LOAD.



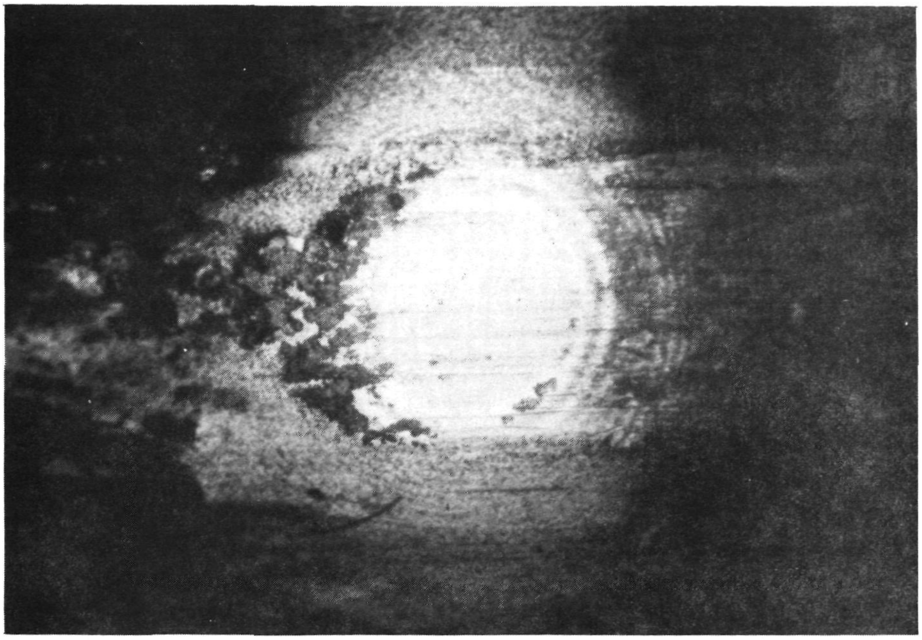
(b) 0.5% MEDIUM GRADE IN 150 cS OIL, 1 kg LOAD.

Figure 5. - MoS_2 distribution during pure sliding U_1 = 0.0021 m/s, U_2 = 0, $\overline{U}$ = 0.0011 m/s.

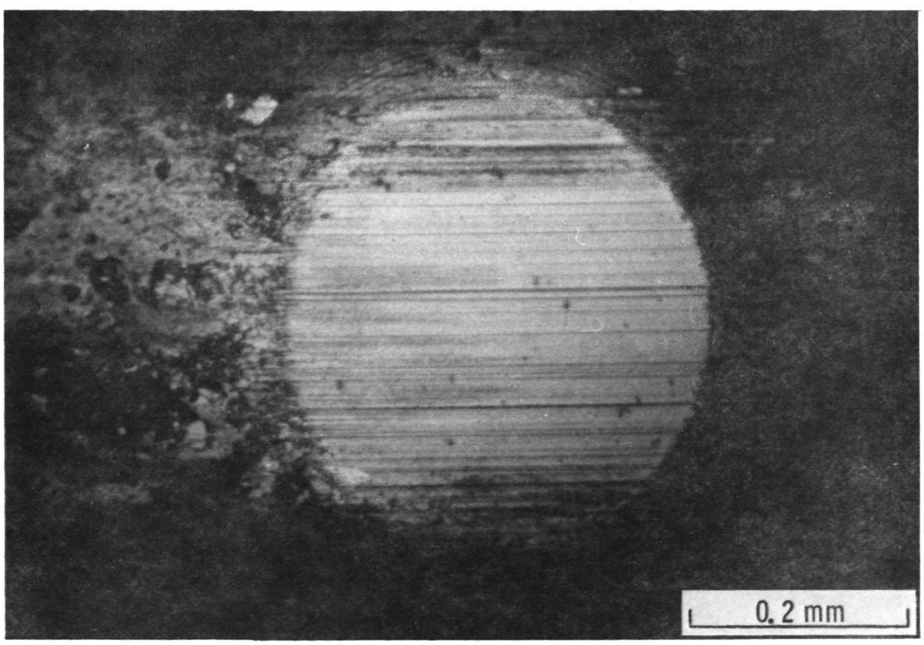


(c) 0.5% COARSE GRADE IN 150 cS OIL, 4 kg LOAD.

Figure 5. - Concluded.



(a) 0.5% SUSPENSION GRADE IN 78 cS OIL, 1 kg LOAD.



(b) 3% COARSE GRADE IN 150 cS OIL, 4 kg LOAD.

Figure 6. - MoS_2 distribution during pure sliding at a higher sliding velocity than in figure 5. U_1 = 0.0146 m/s, U_2 = 0, $\overline{U}$ = 0.0073 m/s.

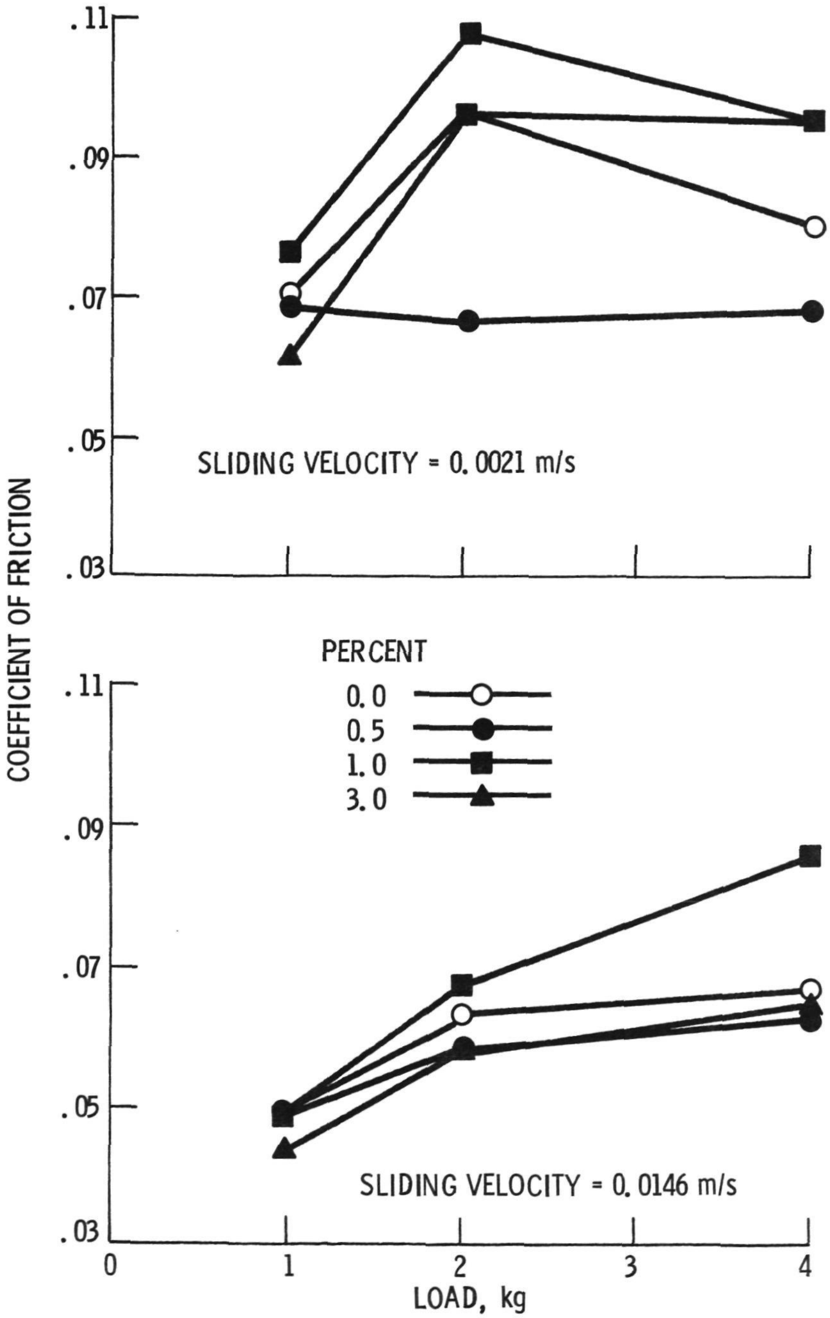
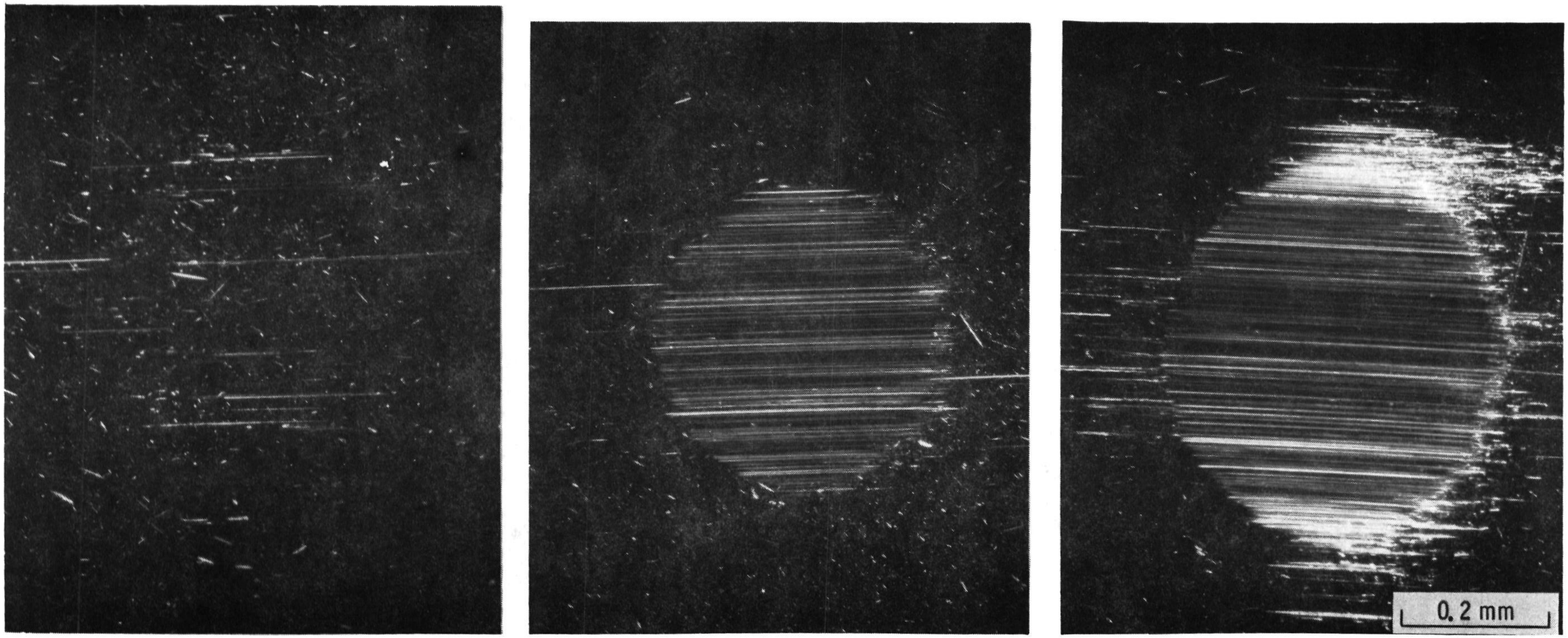


Figure 7. - Sliding friction with suspension grade MoS_2 in 150 cS oil.

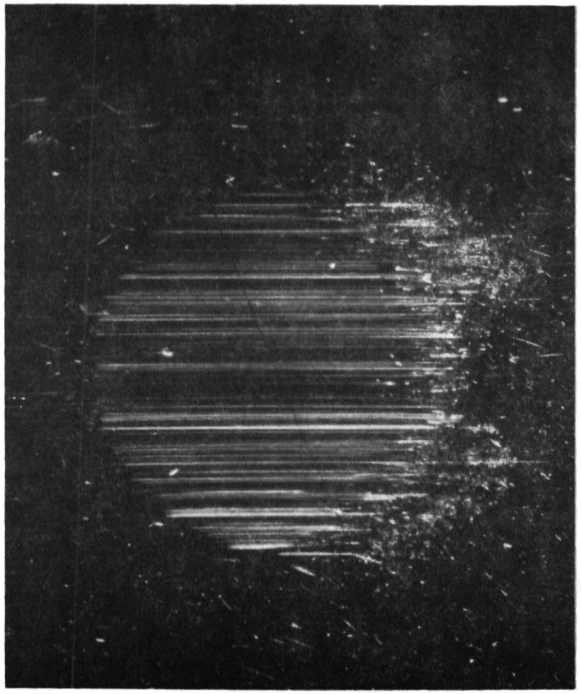


(a) OIL ONLY.

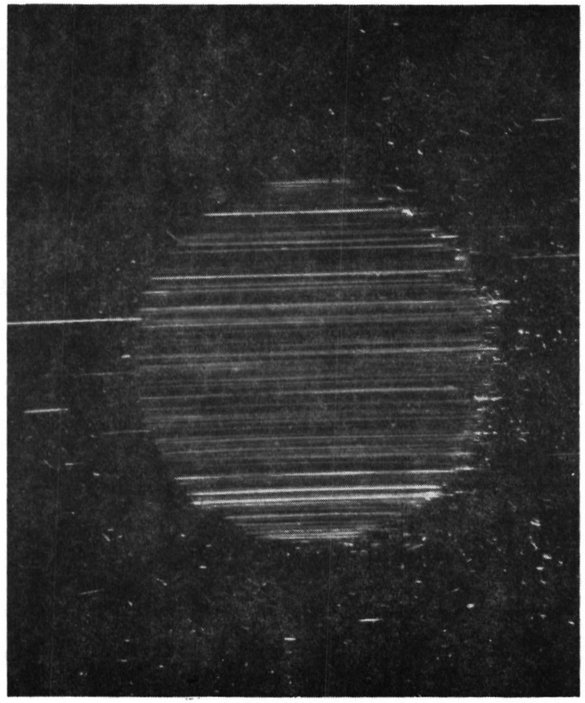
(b) 0.5% MEDIUM GRADE MoS_2.

(c) 0.5% COARSE GRADE MoS_2.

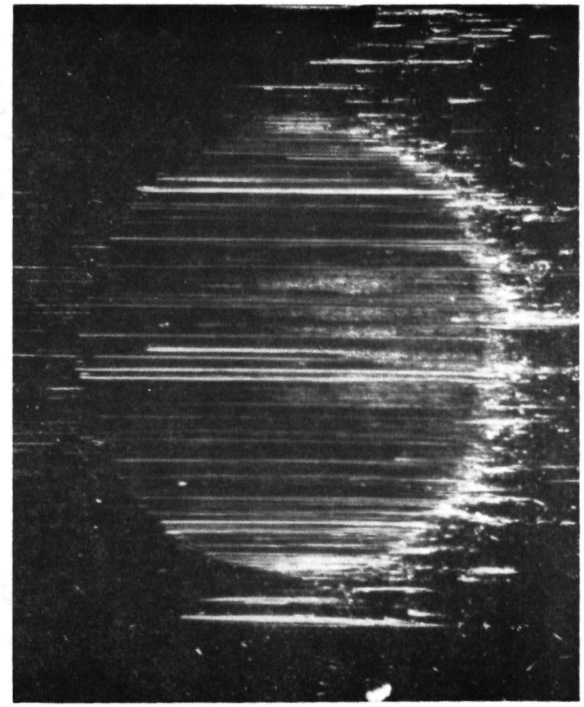
Figure 8. - Effect of MoS_2 in 150 cS oil on abrasion of polished steel ball during sliding contact. 125X original magnification.



(d) 3% SUSPENSION GRADE MoS_2.

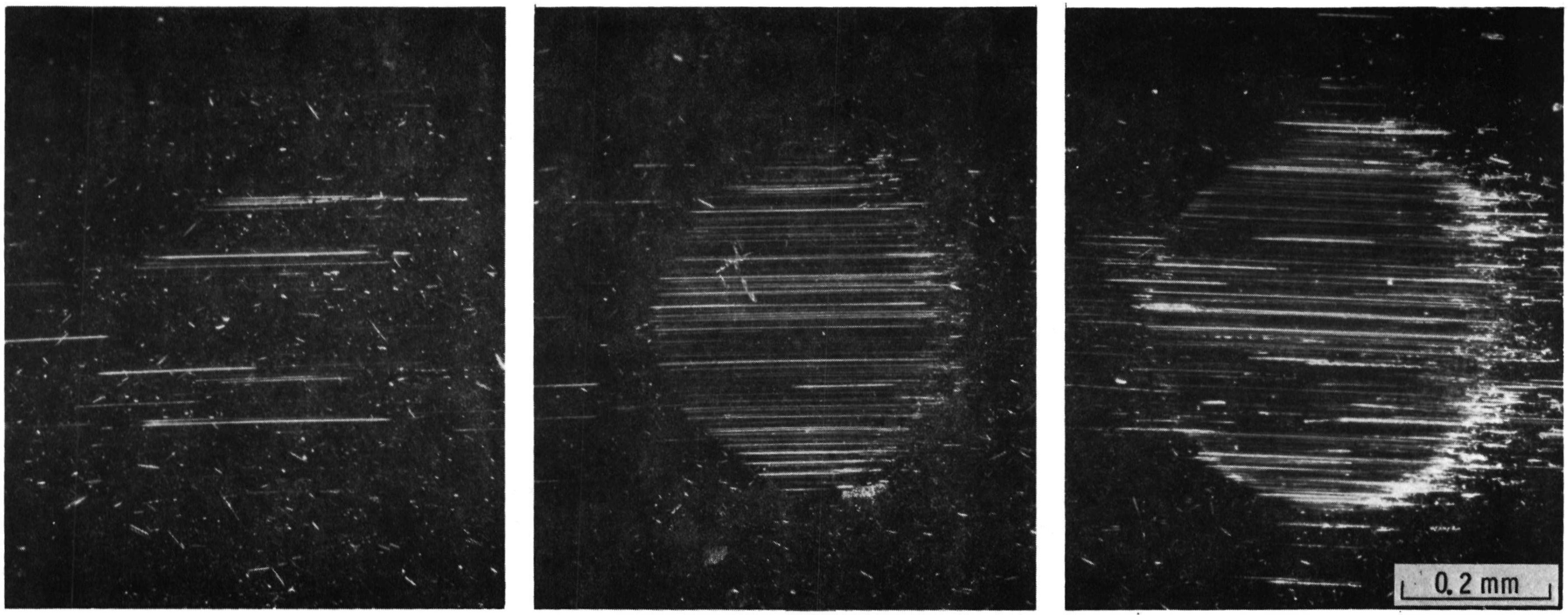


(e) 3% MEDIUM GRADE MoS_2



(f) 3% COARSE GRADE MoS_2.

Figure 8. - Concluded.



(a) OIL ONLY. (b) 0.5% SUSPENSION GRADE MoS_2. (c) 0.5% COARSE GRADE MoS_2.

Figure 9. - Effect of MoS_2 in 78 cS oil on abrasion of polished steel ball during sliding contact. 125X original magnification.

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15. Supplementary Notes

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16. Abstract

The dynamics of MoS_2 particles in a mineral oil dispersion are studied in the same manner as reported in Part I for graphite dispersions. A Hertzian contact consisting of a steel ball in contact with a glass disk is lubricated with MoS_2 dispersions and observed by optical microscopy at various slide/roll conditions. In general the behavior of MoS_2 and graphite are similar. That is, the solids tend to enter the contact and form a film on the contacting surfaces whenever a rolling component of motion is used, but solid particles seldom enter the contact during pure sliding. MoS_2 has more pronounced plastic flow behavior than graphite. However, the polished steel ball is more readily scratched by MoS_2 than by graphite. Under the conditions of these studies, lower friction and wear are observed with pure oil rather than with the dispersions. However under other conditions (such as different contact geometry or rougher surfaces) the solid lubricant dispersions might be beneficial.

17. Key Words (Suggested by Author(s)) Solid lubricant dispersions; Molybdenum disulfide; Dispersion lubricating mechanisms; Lubricant particle dynamics	18. Distribution Statement Unclassified - unlimited; STAR Category 27

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