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FRICTION AND WEAR INVESTIGATION OF MOLYBDENUM DISULFIDE

II - EFFECTS OF CONTAMINANTS AND METHOD OF APPLICATION

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SUMMARY

An experimental study was conducted using a low-speed kinetic-friction apparatus to show the effects of method of application and contaminants on the lubricating characteristics of molybdenum disulfide MoS_2.

Friction coefficients for a purified grade of MoS_2 can vary from 0.025 to 0.12 merely by changing the method of application. Contaminants present in commercial grades of MoS_2 do not increase friction but can (e.g., 0.5 percent silica) adversely affect wear.

In room atmosphere small amounts of oil (5 to 10 percent) reduce friction below that obtained with either purified MoS_2 or with oil alone. At least 10 percent MoS_2 should be present in the oil to obtain the lowest friction. The lowest friction values were obtained at the highest temperatures used (300° F) where the adverse effects of moisture were minimized.

INTRODUCTION

The effective use of molybdenum disulfide MoS_2 is dependent on the ability to supply and to maintain a solid lubricant film between surfaces. The bonded film method has been studied in more detail than other methods of application and some data indicate that bonding is a more effective method of application than the use of loose powders and fluid mixtures (refs. 1 to 4). The various methods of application therefore merit further study.

The friction and wear properties of many lubricants are a function of the impurities they contain. Examples of beneficial contaminants are the presence of absorbed gas or moisture in graphite (refs. 5 and 6) and polar compounds in oils (ref. 7). Reference 8 shows that moisture from humid air was harmful to lubrication by MoS_2.

Impurities are present in molybdenite as mined and the processes for the removal of natural impurities (gangue, which is mostly silica) introduce oil and water and possibly some residual acids as contaminants. Abrasive molybdenum trioxide MoO_3 (ref. 9) is probably formed during purification (by heating to remove oil and water); and grinding processes and high surface temperatures of sliding may form additional oxides. The method of use may also introduce contamination, such as the carrier vehicle (oils, water, greases, or volatile fluids), and other contaminants, such as metallic wear debris, abrasives from the atmosphere, and products of fuel combustion in engines, are introduced from the operating environment. If small quantities of impurities are not harmful in lubrication, the more readily available (and less expensive) impure grades could be used and the method of application would not be critical in that regard.

A series of experiments was conducted at the NACA Lewis laboratory to determine the friction and wear properties of surfaces lubricated by MoS_2. The objects of the experiments were to study the effects of applying the MoS_2 by different methods and also the effects of small amounts of contaminants (abrasive, water, and oil). This study was conducted with a low-speed kinetic-friction apparatus using steel specimens.

APPARATUS AND PROCEDURE

Apparatus

The apparatus used in this investigation is shown in figure 1 and described in detail in reference 8. The rotation specimen (fig. 2) was a 2-inch-diameter ring of nickel-molybdenum steel (SAE 4620) case hardened to Rockwell C-62. Three nodes (2-in. radius) were ground on the edge of the test ring that rotated at 11 rpm (5.7 ft/min) against the disk surface. Material removed from the contacting area of the node by grinding was not sufficient to affect surface hardness. The surfaces of the nodes were polished to remove grinding marks. The disk specimens (SAE 1020 steel) were finished by vapor blasting and had a surface roughness of 60 rms as measured with a profilometer. The vapor-blast finish was used to avoid the directional surface roughness of ground surfaces which make it difficult to maintain a lubricant film (ref. 8).

The lower assembly of the apparatus contained an electrical resistance heater used for runs at temperatures to 300° F. A thermocouple was mounted on the surface at the center of the plate that supported the disk specimen.

Procedure

Cleaning procedure. - Prior to each run both friction specimens were cleaned according to the following procedure:

(1) Washed in 50/50 acetone-benzene solution

(2) Scrubbed with repeated applications of moist levigated alumina

(3) Washed in tap water

(4) Washed in distilled water

(5) Washed in 95-percent alcohol

(6) Dried in a stream of warm air and stored in a desiccator

Preparation of lubricants. - The approximate composition as obtained from the specification of the various commercial grades of MoS_2 are given in table I. The most highly purified grade was used as the basic material for the runs in which contaminants were added. Silica (technical grade, 200 mesh) was added as percentage by weight to the MoS_2 and then mixed carefully using a spatula. Distilled water and a low-boiling-point distillation cut of a commercial medicinal white oil were used as the other contaminants. In order to obtain good distribution of oil in the powder for runs with low oil concentration (under 10 percent oil), benzene was used as a dispersing solvent. After thorough mixing, the benzene was removed by evaporation in a continuous supply of clean dry air; the oil and MoS_2 mixture was further dried in an oven at 150° F for 8 hours.

Test procedure. - In all cases the MoS_2 (powder or bonded film) was applied to the disk specimen. A new slider and disk specimen were used for each run after finishing and cleaning as previously described. Approximately 20 grams of the solid lubricant were used in each run. Unless specifically noted, a load of 40 pounds was used in all runs. The tests were begun and run to completion without interruption; friction force was continually observed and measurements were recorded approximately every 10 minutes. The runs in dry air (more fully described in ref. 8) were preceded by a 1/2-hour drying period during which time, as well as during the test, dry room-temperature air (from a -70° F refrigerated air source) flowed through the enclosing chamber. For the runs at 300° F, the specimens and lubricants were held at the test temperature for 1/2 hour before the runs. Friction runs were of 1- or 2-hour duration, and wear runs were 6 hours long. In special runs MoS_2 was returned to the wear track by the guide vanes shown in figure 3. The amount of wear was determined with a planimeter by measuring areas of wear spots from projected enlarged images of the wear areas on the slider specimens. At the end of each run, all specimens were examined microscopically and photographed.

RESULTS AND DISCUSSION

The results are presented with reference to two phases of the investigation. The data concerned with the effect of methods of application on the lubricating ability of MoS_2 of a highly purified commercial grade (grade A, table I) are discussed first. Secondly, the effects of contaminants on the lubrication properties of MoS_2 are reported. Many of the points to be discussed in this section are based on visual observations of the specimen surfaces after test.

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METHODS OF APPLICATION

The effect of running time on friction of steel surfaces lubricated with MoS_2 applied by various methods is shown in figure 4. Friction coefficients for a purified grade of MoS_2 varied from 0.025 to 0.12 by changing the method of application. Study of the surfaces in numerous runs has indicated that an initial run-in period occurred. During that interval the powder was worked into a film on the disk and well-defined areas of shear were established on the nodes of the specimens. As discussed in reference 8, the MoS_2 is considered to flow plastically under load to fill the surface interstices and, therefore, the apparent area of contact (the entire geometrical area of contact) may be the area of shear.

In runs with minute amounts of MoS_2 deposited from a volatile solvent or by dusting with lens tissue, there was insufficient MoS_2 present to fill the interstices between the surfaces. Therefore, the shear area could not approach the apparent area in contact and friction was very low (fig. 4). The film formed by light dusting or rubbing with lens tissue was more tenacious than that obtained using a volatile fluid carrier. The film obtained from the volatile carrier did not completely prevent metallic contact, which probably explains the slight increase in friction with time during the latter part of the run.

It is significant that very small amounts of MoS_2 can prevent surface failure and maintain very low friction. With MoS_2 supplied from a volatile fluid, minimum friction could be maintained by continually replenishing the MoS_2 at a rate sufficient to prevent harmful metallic contact but insufficient to form large shear areas.

The other methods of application provided sufficient MoS_2 to form a continuous shear area. After the run-in period, essentially constant friction was obtained with: (1) excess MoS_2 loose power, (2) excess loose powder returned to the wear track by guide vanes, and (3) a commercial bonded film. The differences in friction values for these methods of application may have been caused by variations in effective shear strengths of the various films as a result of such factors as orientation and presence of extraneous material.

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When the powdered MoS_2 was continually returned to the sliding area on the disk by means of the guide vanes shown in figure 3, there was no apparent run-in period and the friction remained essentially unchanged for the duration of the 2-hour run (fig. 4). The continued introduction of fresh powder into the shear area resulted in a lesser degree of surface orientation than would be experienced with an established film that was continually subject to repeated shear in the same direction. It is probable that orientation was one of the primary effects of the run-in period with most films. In that case, where the film surface did not have a chance to become oriented, friction remained constant and somewhat higher than in runs without the guide vanes. Lack of orientation could increase effective shear strength and also reduce plastic flow of the lubricant film. It should be noted that the force necessary to return loose MoS_2 to the wear track was part of the measured friction value but was insignificant in regard to its effect on friction coefficients.

The run with a commercially bonded film of MoS_2 (fig. 4) showed final friction values slightly higher than those obtained with an excess amount of loose powder. The usual run-in period was observed. The commercial mixture used to form these films was composed of 15 percent by weight of resinous bonding material (Epon). It is reasonable to assume that the bonding resin may have caused the slightly increased friction through its effect on over-all shear strength of the surface film.

The effect of humidity is a very important variable with regard to lubrication by MoS_2; and this point is discussed in detail in reference 8, which shows that reducing humidity improves lubricating effectiveness. Comparison of data from preliminary runs at room humidity (approximately 40 to 50 percent relative humidity) with those reported herein indicates that the return of loose powder to the shear area by guide vanes was the method of application least affected by change in humidity. With all methods of application, friction at room humidity was higher than that obtained in dry air.

The data of figure 5 obtained with an excess amount of MoS_2 show that friction coefficient did not change significantly with increasing load. In reference 10, where a greater load range is covered, a decreasing trend of friction coefficient with increasing loads is observed.

Contaminants

The data of figure 6 were obtained with various commercial grades of MoS_2. The principal contaminants in the various grades described in table I are gangue (mostly silica), oil, and water. There is only a small difference in friction with grades A and B where the composition variable is one of gangue (silica) content. The less pure grades (C and D, table I) resulted in lower friction than was obtained with the most highly purified grade. This difference appears to be associated with oil and water, which are present in the least pure grades. Study of the disk specimens showed that with the water and oil contaminants present the MoS_2 spalled out of the voids between the contacting asperities with the result that the amount of surface sheared was reduced. The reduction in shear area would be accompanied by a reduction in friction. 2917

Although the data of figure 6 indicate that friction is not adversely affected by the various contaminants in commercial grades of MoS_2, consideration of the known characteristics of the contaminants introduces concern about their effect on wear. In particular, the silica present in gangue could accelerate wear by its abrasive action. Also, the data of reference 8 indicate that the presence of moisture either from humid air or added water increases wear and can also increase friction.

In order to determine the effect of silica on wear and friction, it was necessary to eliminate the adverse effect of moisture as an experimental variable. Therefore, the series of runs to obtain the data of figure 7 was made in dry air. These runs were made with silica (technical grade, 200 mesh) additions to purified MoS_2 (grade A). The data show that both wear and friction increase with greater silica content in MoS_2. Wear obtained with MoS_2 alone was insignificant compared with the wear obtained with 0.5 percent or more silica in purified MoS_2. The characteristics (particle size, etc.) of the silica naturally contained in MoS_2 may not correspond to the silica used in these experiments and, therefore, any quantitative use of these data must be made with caution. It is, however, of importance to point out that except for grade A shown in table I all the commercial grades listed contain over 0.5 percent gangue.

At room humidity, wear and friction were decreased by the presence of small amounts of oil in MoS_2. In dry air with no oil present, the wear of steel surfaces lubricated with purified MoS_2 was less than in room-humidity runs with mineral-oil contamination of the MoS_2. This effect of oil is one of minimizing the adverse influence of moisture on wear. When moisture effects were essentially eliminated by making runs in dry air, 2-hour friction experiments were too short in duration to show any measurable effect on wear of contamination of MoS_2 by oil.

The data of figure 8 show that the friction for an excess of powdered MoS_2 (grade A) decreased with increasing temperatures up to approximately 200° F and that friction was essentially independent of temperature from 200° to 300° F. Previously reported research (ref. 8) shows that friction increases with humidity up to approximately 60 percent relative humidity and then decreases with further increase in humidity. The change in friction with relative humidities of less than 60 percent was attributed to increased metallic contact which accompanied increased humidity. It was considered probable, therefore, that the decrease in friction as temperatures were increased to 200° F was a function of decreased metallic contact resulting from vaporization (desorption) of moisture from the lubricated surface. Data obtained at low humidity tend to substantiate that point. At low humidity there was very little change in friction with increasing temperatures; this would indicate that reduction in shear strength is insufficient to account for the reduction in friction obtained with increasing temperatures in room atmosphere. The friction coefficients obtained at low humidity were similar to those obtained at the higher temperatures (above 200° F) in room atmosphere.

Additional experiments were made to illustrate further the effect of temperature on lubrication by MoS_2 containing the separate contaminants. Figure 9 presents friction data at room temperature and at 300° F in room atmosphere for excess powdered (grade A) MoS_2, for MoS_2 to which 10 percent water had been added, and for MoS_2 to which 10 percent light mineral oil had been added. The friction of the purified grade of MoS_2 at 300° F was unaffected by running time after the first 10 minutes of the run. Continued sliding for periods up to 6 hours failed to show any change in friction or tendency toward lubrication failure. Figure 10 is a photomicrograph of a MoS_2 film after a run at room temperature and atmosphere; the surface shows small metallic contacts protruding through the MoS_2 film in a manner characteristic of the way in which partial failure of MoS_2 lubrication begins. After the run at 300° F, no metallic contacts were visible through the film.

At room temperature, the presence of 10 percent water decreases the friction (fig. 9(b)) below that obtained with purified MoS_2 alone (fig. 9(a)). One effect of water additions to MoS_2 was shown in reference 8 to be that of decreasing shear area by preventing the formation of a continuous film. The initial low friction (fig. 9(b)) is considered to result from small shear area, and the increasing friction trend as the run progressed is associated with increased metallic contact. At 300° F, as might be expected, the 10 percent water addition was caused to evaporate and the friction data obtained were approximately the same as those for the uncontaminated MoS_2 at 300° F (fig. 9(a)).

With 10 percent light mineral oil present as a contaminant in MoS_2, low friction values were obtained for the duration of the runs at both room temperature and at 300° F (fig. 9(c)). At room temperature, friction continued to increase slightly with running time for all except the initial 2 minutes of the run. With continued operation beyond the 2-hour period, friction coefficients closely approached the value (0.16) for mineral oil alone shown in figure 11(b). At 300° F, the same low friction coefficient (0.05) was obtained for all but the first few minutes of the entire run. The friction trends and values at 300° F are essentially the same regardless of whether the MoS_2 had been contaminated with oil or water or had not been contaminated.

A series of data is presented in figure 11 to show the effect of various weight percentages of oil and MoS_2 on friction coefficients. These data were obtained with mixtures and not with stable suspensions. This type of experiment was not necessarily penalized by the instability of the mixture, since any solids that settle out would be deposited on the disk surface where they could contribute to lubricating effectiveness.

Figure 11(c) summarizes the data given in the preceding parts of the figure. This curve shows more directly the effect of various weight percentages of oil in MoS_2 on coefficient of friction. Additions of oil up to 10 percent by weight to MoS_2 cause a very marked reduction in friction. It may be seen from figures 11(a) and (b) that oil concentrations of 10 percent or more are necessary to obtain stable values over extended periods of time. With oil concentrations from 10 to 90 percent, friction was essentially unchanged. These data give an indication of the reduction in friction that can be obtained with mixtures (or suspensions) of MoS_2 and mineral oil but should not be interpreted to imply absolute limits for effective concentrations. Limiting concentrations will probably vary with design considerations for each lubrication problem. With the higher percentages of oil (above 90 percent), friction values increase to that of the oil alone.

The use of oil mixtures can be a convenient way of applying MoS_2 in many cases. The effectiveness of mixtures of MoS_2 and oil, however, depends on how well the MoS_2 particles are trapped between the contacting surfaces. The data of figure 11 were obtained with a cylindrical surface sliding on a flat surface. The action of MoS_2 particles in oil during lubrication of sliding surfaces was studied further by placing a sample of oil containing MoS_2 on a glass microscope slide and watching the behavior of the MoS_2 particles through a microscope as the fixed lubricated glass surface was traversed by a lightly loaded reciprocating steel ball. This type of experiment showed that the oil promoted movement of the MoS_2 away from the area of contact and, hence, the lubricating effectiveness of the MoS_2 was impared. It was observed that the MoS_2 particles did not remain in the contact area between the ball and the glass surface unless trapped there by surface roughness of the ball or by a large accumulation of solid particles. It is therefore impossible to generalize on the effectiveness of oil as a carrier for MoS_2 on the basis of studies of only one type of surface configuration. Shape of sliders, surface roughness, and high MoS_2 concentrations can be used to improve lubricating effectiveness of mixtures of oil and MoS_2.

SUMMARY OF RESULTS

Under conditions of the experiments reported herein, the following characteristics of molybdenum disulfide MoS_2 as a solid lubricant for steel on steel were observed:

1. Friction coefficients for a purified grade of MoS_2 varied from 0.025 to 0.12 merely by changing method of application.

2. Impurities found in commercial grades of MoS_2 did not have any harmful effect on friction but can adversely influence wear. Silica, which is a common impurity, caused appreciably increased wear when it was present in concentrations as low as 0.5 weight percent.

3. In room atmosphere, contamination of MoS_2 with small amounts (5 to 10 weight percent) of mineral oil provided much lower friction and less severe wear than could be obtained with either a purified grade of MoS_2 or mineral oil alone. Friction increased for mixtures containing more than 90 percent oil but with oil concentrations from 10 to 90 percent friction was essentially unchanged.

4. The friction and wear properties of MoS_2 at 300° F were superior to those of the same material at room temperature.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 8, 1953

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TABLE I. - APPROXIMATE COMPOSITION OF VARIOUS COMMERCIAL GRADES OF MOLYBDENUM DISULFIDE

[Data obtained from a Climax Molybdenum Company bulletin; values given are percent by weight.]

Composition	Purified grades of concentrate			Mill concentrate
	A	B	C	D
Molybdenum disulfide	99.9	99	90-94	78-88
Gangue (usually SiO_2)	0.1 (mainly iron oxide)	0.7	0.7	3.0 to 12.5
Mineral oil	------------	0.3 (max)	3.0 to 5.0	5.0
Water	------------	0.2 (max)	3.0 to 5.0	5.0
Copper	------------	-----	----------	0.5 (max)

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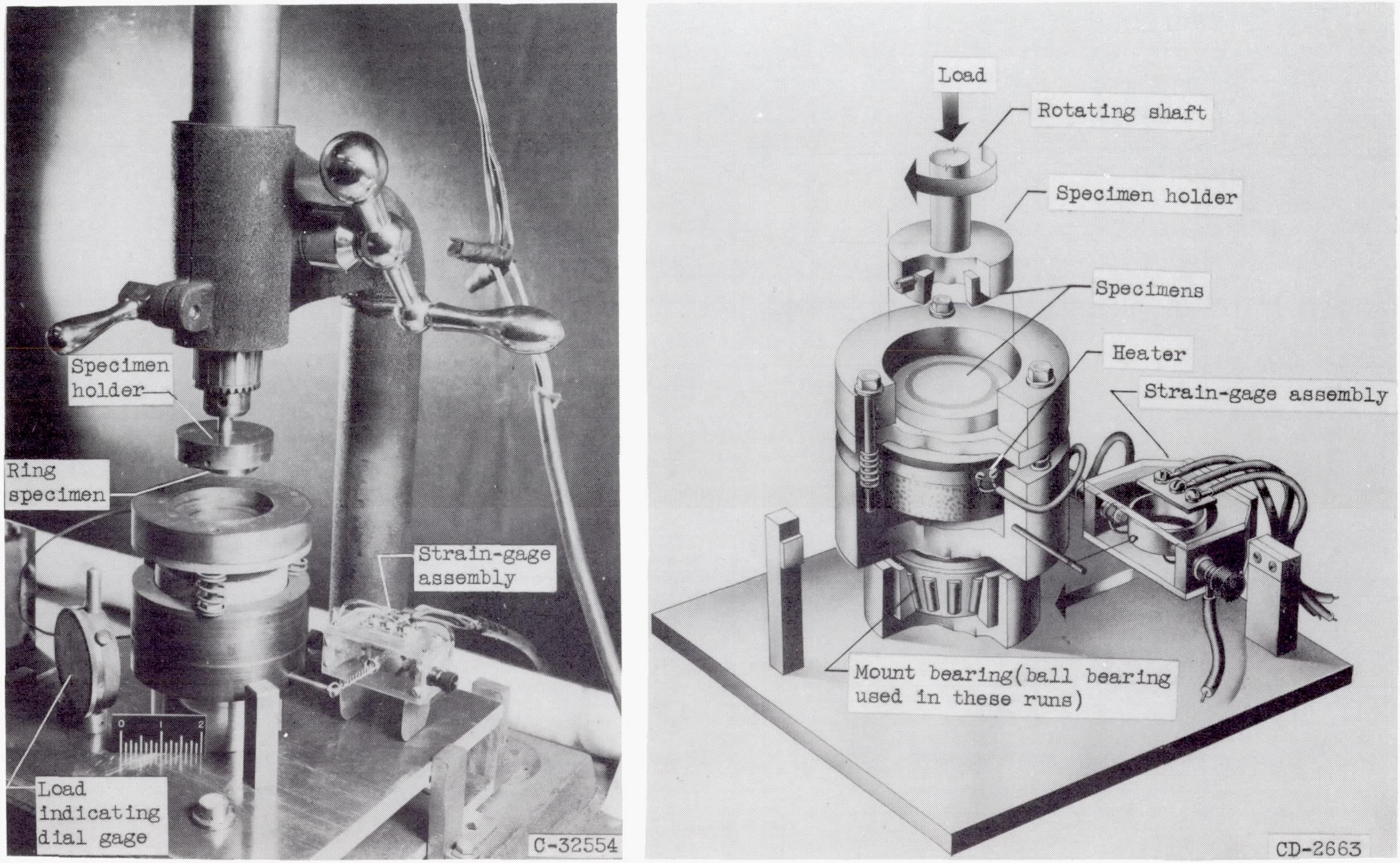

(a) Photograph.

(b) Diagrammatic sketch.

Figure 1. - Detail of basic elements of kinetic-friction apparatus.

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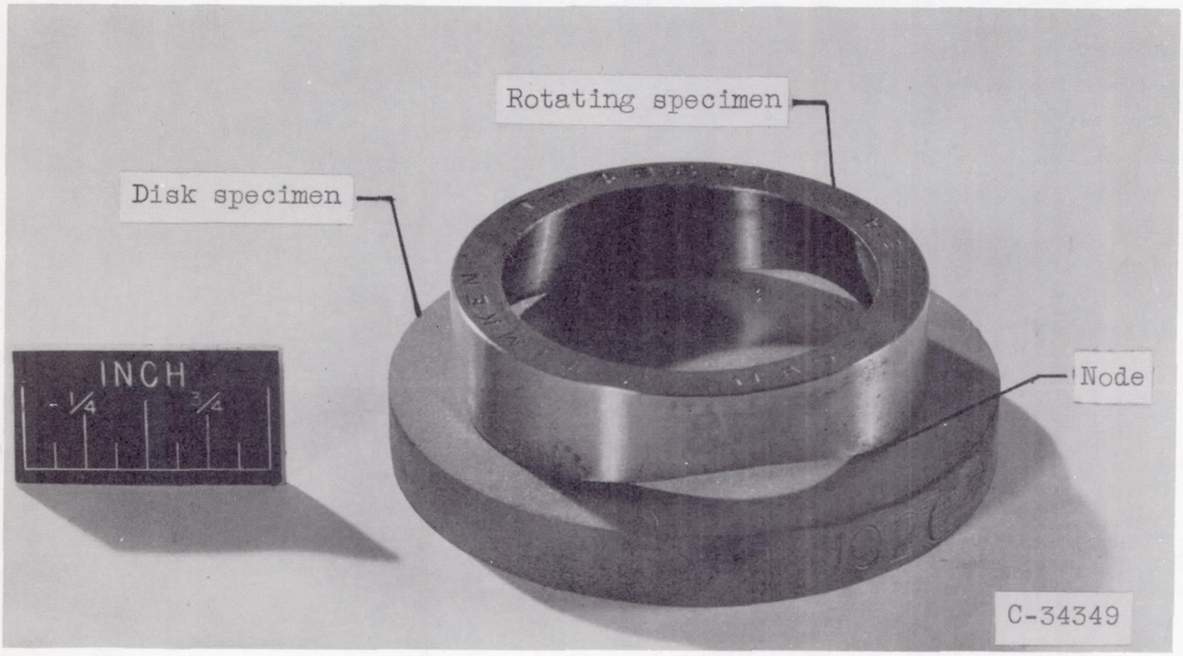

Figure 2. - Friction specimens.

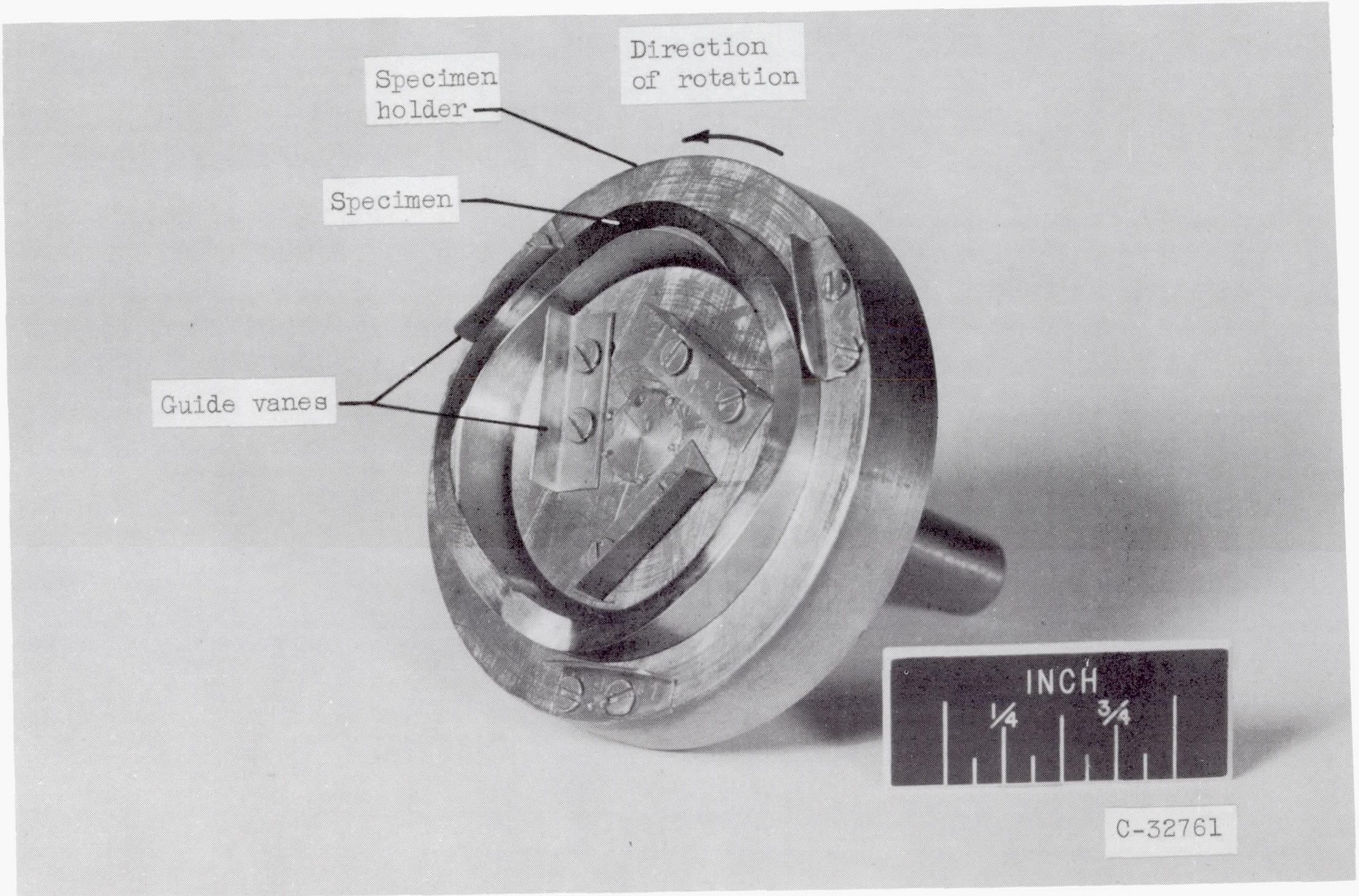

Figure 3. - Rider-holder assembly with guide vanes.

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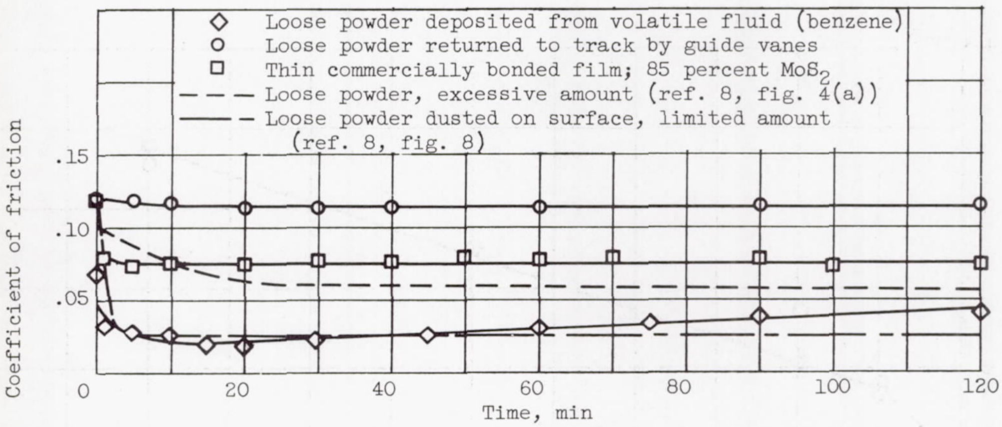


Figure 4. - Effect of method of application on coefficient of friction of steel surfaces lubricated by purified MoS_2 in dry air (below 6 percent relative humidity). Load, 40 pounds; sliding velocity, 5.7 feet per minute.

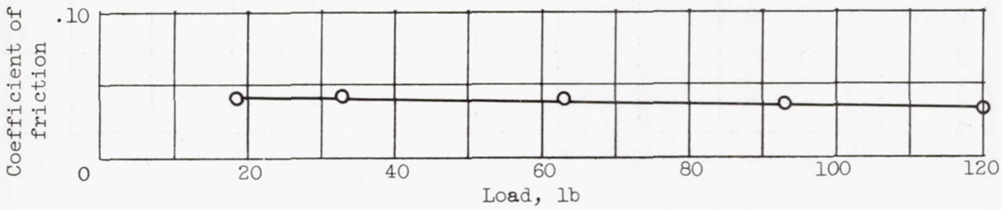


Figure 5. - Effect of load on coefficient of friction of steel surfaces lubricated with excess of loose powdered purified MoS_2 in dry air. Sliding velocity, 5.7 feet per minute.

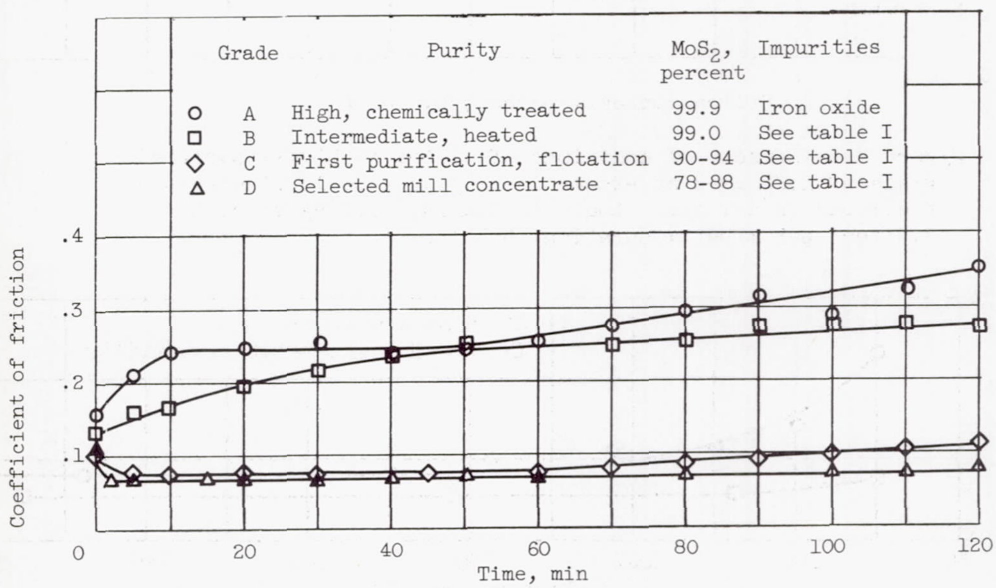


Figure 6. - Effect of various commercial grades of MoS_2 on coefficient of friction using an excess of loose powder. Load, 40 pounds; sliding velocity, 5.7 feet per minute; room atmosphere (40 to 50 percent relative humidity).

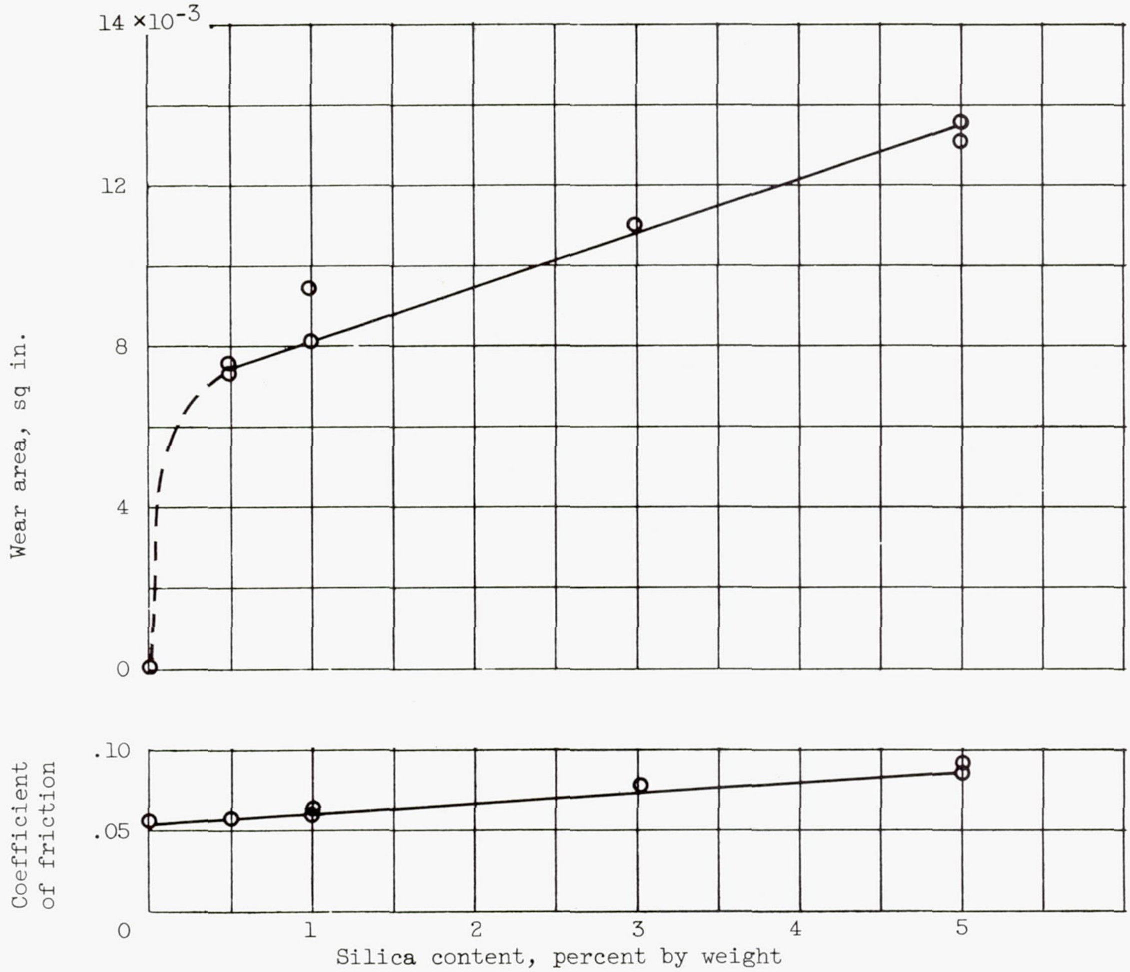


Figure 7. - Effects of additions of silica to highly purified grade of MoS_2 used as excess loose powder to lubricate steel specimens in dry air. Load, 40 pounds; sliding velocity, 5.7 feet per minute; duration, 6 hours.

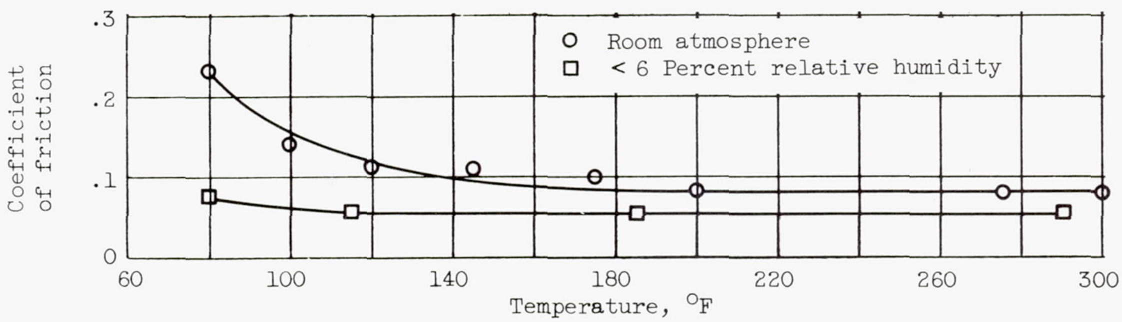
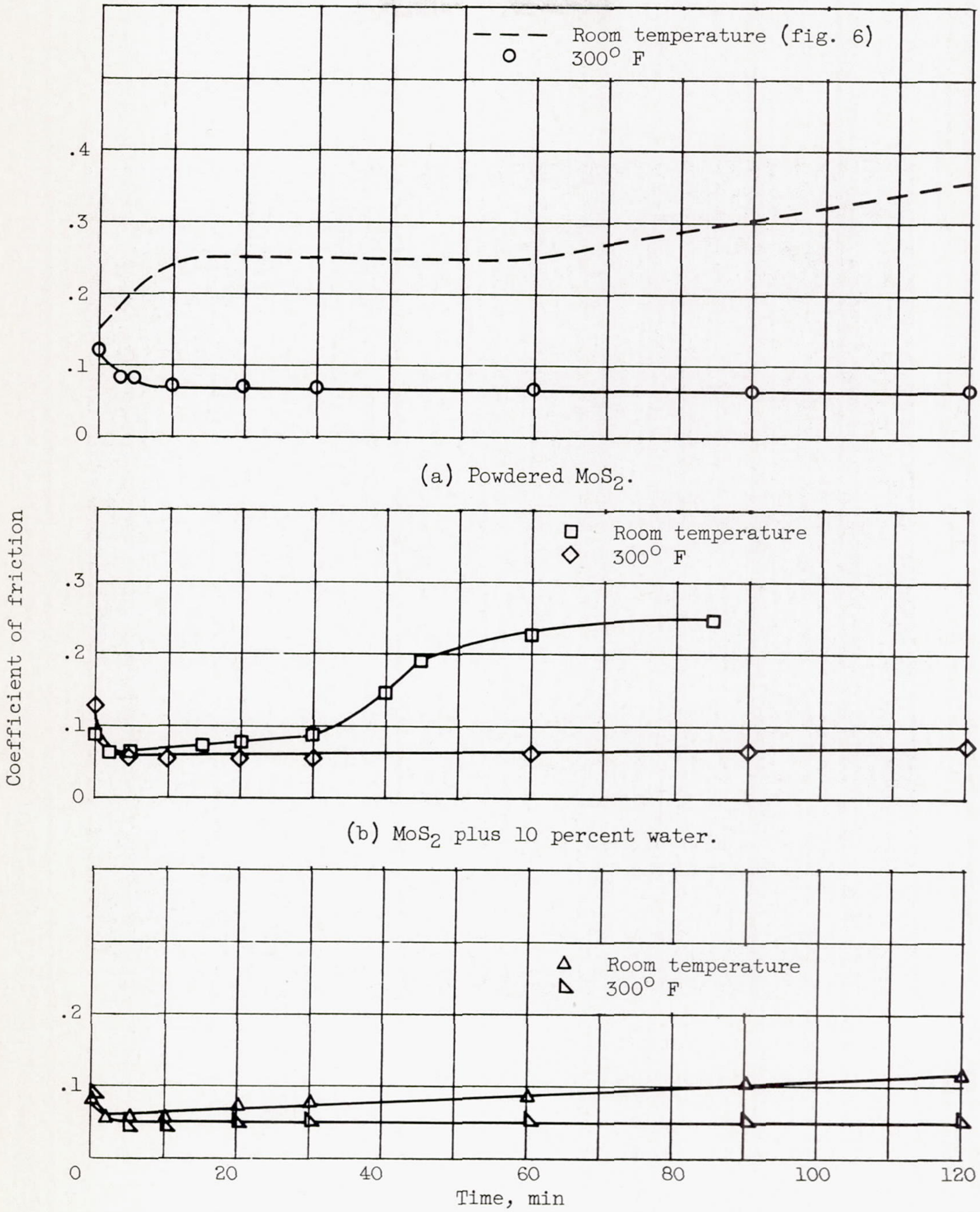


Figure 8. - Effect of temperature on coefficient of friction of steel surfaces lubricated with an excess of loose purified MoS_2 (grade A, table I). Load, 40 pounds; sliding velocity, 5.7 feet per minute.



(c) MoS_2 plus 10 percent oil (low-boiling-point cut of medicinal white oil).

Figure 9. - Effect of running time on coefficient of friction of purified loose MoS_2 powder with no contamination, with 10 percent water, and with 10 percent light mineral oil at room temperature and at 300° F. Load, 40 pounds; sliding velocity, 5.7 feet per minute; room atmosphere (40-50 percent relative humidity).

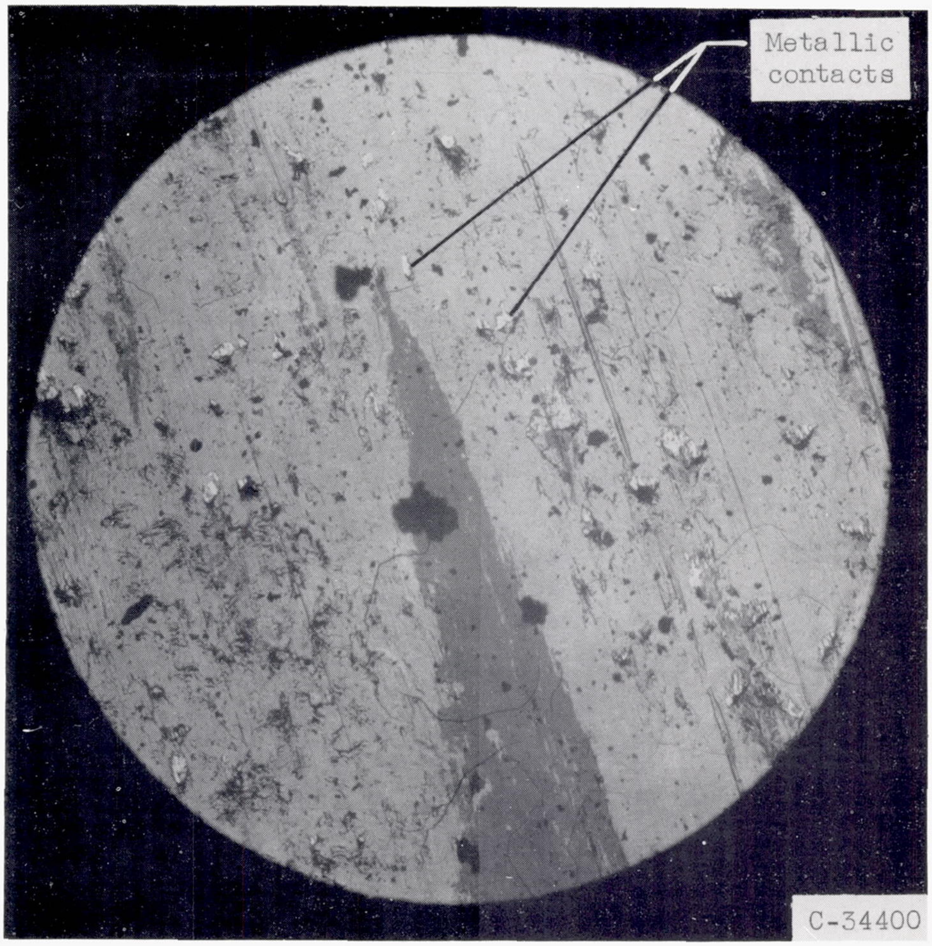
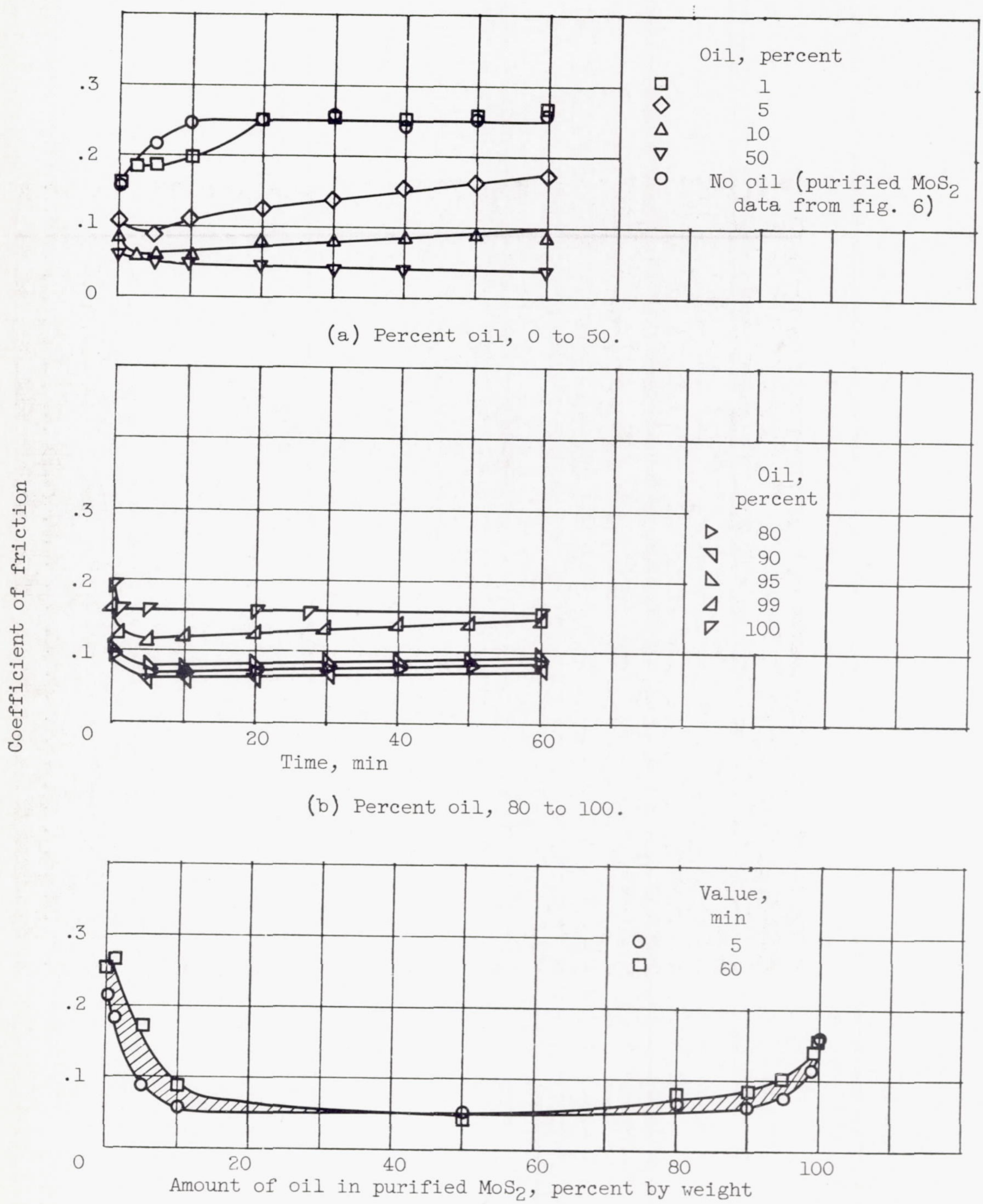


Figure 10. - Photomicrograph of MoS_2 film on disk surface showing small metallic contacts protruding through film. X100.



(a) Percent oil, 0 to 50.

(b) Percent oil, 80 to 100.

(c) Replot of data from figures 11(a) and (b).

Figure 11. - Effect of oil and MoS_2 mixtures on coefficients of friction for various periods of times. Load, 40 pounds; sliding velocity, 5.7 feet per minute; room atmosphere (40-50 percent relative humidity) and temperature. Oil was a low-boiling-point distillation cut of a commercial medicinal white oil.