# REPORT 1254

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# REPORT 1254

## FRICTION, WEAR, AND SURFACE DAMAGE OF METALS AS AFFECTED BY SOLID SURFACE FILMS[1]

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### SUMMARY

*As predicted by friction theory, experiments showed that friction and surface damage of metals can be reduced by solid surface films. The ability of materials to form surface films that prevent welding was a very important factor in wear of dry and boundary lubricated surfaces. Films of graphitic carbon on cast irons, NiO on nickel alloys, and FeO and $Fe_3O_4$ on ferrous materials were found to be beneficial. Abrasive films such as $Fe_2O_3$ or $MoO_3$ were definitely detrimental. It appears that the importance of oxide films to friction and wear processes has not been fully appreciated.*

*The chemical reaction films FeS and $FeCl_2$ were both effective in the prevention of surface welding. Because of its lower shear strength, $FeCl_2$ was more effective than FeS in reducing friction.*

*Solid lubricants such as molybdenum disulfide and graphite warrant careful consideration for use under severe conditions such as high temperatures. Surface films of those materials were found to have beneficial results in friction studies at high loads and high sliding velocities. Bonding mechanism studies showed that both $MoS_2$ and graphite can be bonded to many surfaces by use of a resin-forming liquid. Rolling contact bearing experiments showed that effective lubrication could be maintained at temperatures of 1000° F by injection of minute amounts (0.001 lb/min) of these solids. In the presence of oxygen, $MoS_2$ oxidizes to $MoO_3$ at a very low rate at 750° F, the process becoming rapid at temperatures above 1050° F.*


### INTRODUCTION

This paper is based on NACA research data obtained during a series of investigations in the field of lubrication, friction, and wear conducted from 1946 to 1954. Each investigation in the series had specific objectives, but all were directed toward determination of the role of surface films in friction and wear.

As is well known, the presence of a contaminating film between sliding surfaces can have a marked effect on friction, wear, and surface damage. (A contaminant is defined as any material other than those comprising the sliding surfaces.) Some contaminants are beneficial (lubricants), while others are detrimental (abrasives). Considerable evidence (refs. 1 to 8) indicates the importance of solid surface films to the compatability of sliding surfaces. Physical and chemical surface changes have been associated with satisfactory and with unsatisfactory operation of metallic sliding surfaces (ref. 2). As an example, "run-in" has long been known to be effective in improving the performance and the load-carrying capacity of surfaces. Among the factors that lead to satisfactory performance are the formation and maintenance of certain beneficent solid surface films.

Such solid films are commonly formed on sliding surfaces by use of extreme-pressure additives in lubricants; chemical films of low shear strength or of low melting point are formed by reaction between additives and the sliding surfaces under conditions of extreme pressure and temperature. Theories for the mechanism of extreme pressure lubrication are discussed in references 3 and 8.

A complete study of the mechanism of action of solid surface films and their effect on friction and surface damage to sliding surfaces encompasses a large number of factors. Some of the fundamental factors are discussed in the THEORY section of this report; factors that are important in the use of solid films have been studied experimentally, and the results obtained are presented herein.

The purpose of this report is to show the relation of surface films to the friction, wear, and surface damage of sliding metals. Study of the role of surface films requires consideration of how the film is formed. The film may be (1) formed naturally, (2) preformed, or (3) formed by other methods having practical value.

A modified version of this paper was submitted by E. E. Bisson to the University of Florida, College of Engineering, as a thesis in partial fulfillment of the requirements for the professional degree of Mechanical Engineer, May, 1954.

### THEORY

Analysis of the effects of solid surface films should include a review of the generally accepted theory of dry friction. This theory predicates adhesion between contacting surfaces. (It is of interest to note that, as early as 1785, Coulomb (ref. 9) recognized that "cohesion" between solids was a factor, although he believed "cohesion" contributed a relatively small part of the total friction force.) The adhesion theory of friction (appendix A) was independently advanced by Merchant in this country (ref. 10) and by Bowden and Tabor in England (ref. 8) at about the same time (1940 to 1942).

The adhesion theory of friction predicates the contact of surfaces through asperities; this theory proposes that friction force is equal to the sum of two terms: a shear term and a ploughing or a roughness term. Since the contact of surfaces

[1] Supersedes NACA TN 3444, "Friction, Wear, and Surface Damage of Metals as Affected by Solid Surface Films," by Edmond E. Bisson, Robert L. Johnson, Max A. Swikert, and Douglas Godfrey, 1955.

is through surface asperities, it has been postulated (ref. 8) that plastic flow occurs at the points of contact until the real contact area $A$ is great enough to support the load. The area $A$ then equals $W/p$, where $W$ is load and $p$ is flow pressure (yield strength in compression). The initial surface contact stress under these conditions is not a low value, but is equal to the flow pressure. Pressure welding occurs at these highly stressed contact points, and the welded junctions must shear before relative motion can take place. The force required to shear them is one of the components of the friction force, generally called the "shear" term.

Appendix A shows the development of the equations expressing the adhesion theory by Bowden and Tabor and by Merchant. Under certain conditions (as when the surfaces are quite clean), the shear term may be the more important of the two, and the second term may be neglected. In appendix A, surface contact pressure is represented by Bowden and Tabor as the symbol $p$ for flow pressure and by Merchant as the symbol $H$ for pressure surface hardness. These two pressures are the same.

For cases in which the ploughing term is negligible, coefficient of friction $\mu$ (defined as ratio of friction force to applied load) is shown (appendix A) to be equal to the ratio of shear strength to flow pressure, that is, $\mu=s/p$. Reduction in friction coefficient for this case therefore depends on reduction in the value of ratio of shear strength to yield strength. The ratio can be reduced by: (1) decreasing shear strength, (2) increasing yield strength, or (3) a combination of (1) and (2). Obtaining both conditions in one material is practically impossible because those materials with low shear strength have low yield strength and vice versa. However, by the use of low-shear-strength films (with thicknesses as small as millionths of an inch) on hard base materials, both desirable conditions may be obtained (ref. 8, p. 112). Thus, low shear strength is obtained without appreciable decrease of the yield strength of the combination. The load will thus be supported through the film by the hard base material, while shear occurs within the soft thin film. These low-shear-strength films can be of the following types: chemical reaction products (oxides, chlorides, sulfides), metals, fluid lubricants, and so forth.

Among the properties other than shear strength, flow pressure, and hardness that are important in the action of solid films are: crystalline structure and orientation, chemical composition, chemical activity, and adsorption.

An important function of surface films (frequently neglected in analyses of their action) is to act as a contaminant for the reduction of welding or adhesion between rubbing surfaces; contaminants reduce welding by reducing contact of clean metal to clean metal. The better the bond between film and surface, the greater the resistance to rupture of the film, and, consequently, the greater the protection from welding.

The discussion of this section shows that the basic requirement for reduction of friction, wear, and surface damage of sliding surfaces is the presence of a low-shear-strength contaminant. This contaminant serves two functions, either or both of which may be influential: (1) to reduce the amount of welding at the surface asperities, and (2) to decrease the shear force. In order to carry out the second function, the contaminant must be a material of lower shear strength than the softer of the two materials which are sliding one against the other (appendix A).

## APPARATUS AND PROCEDURE

### APPARATUS

In the investigations reported, five different pieces of apparatus were used. These are identified as A, B, C, D, and E; general descriptions of them are given in the following paragraphs, and detailed descriptions in the references noted. Data presented in this report are identified in the various figures according to the apparatus used.

Apparatus A; for kinetic friction (ref. 11).—Most of the

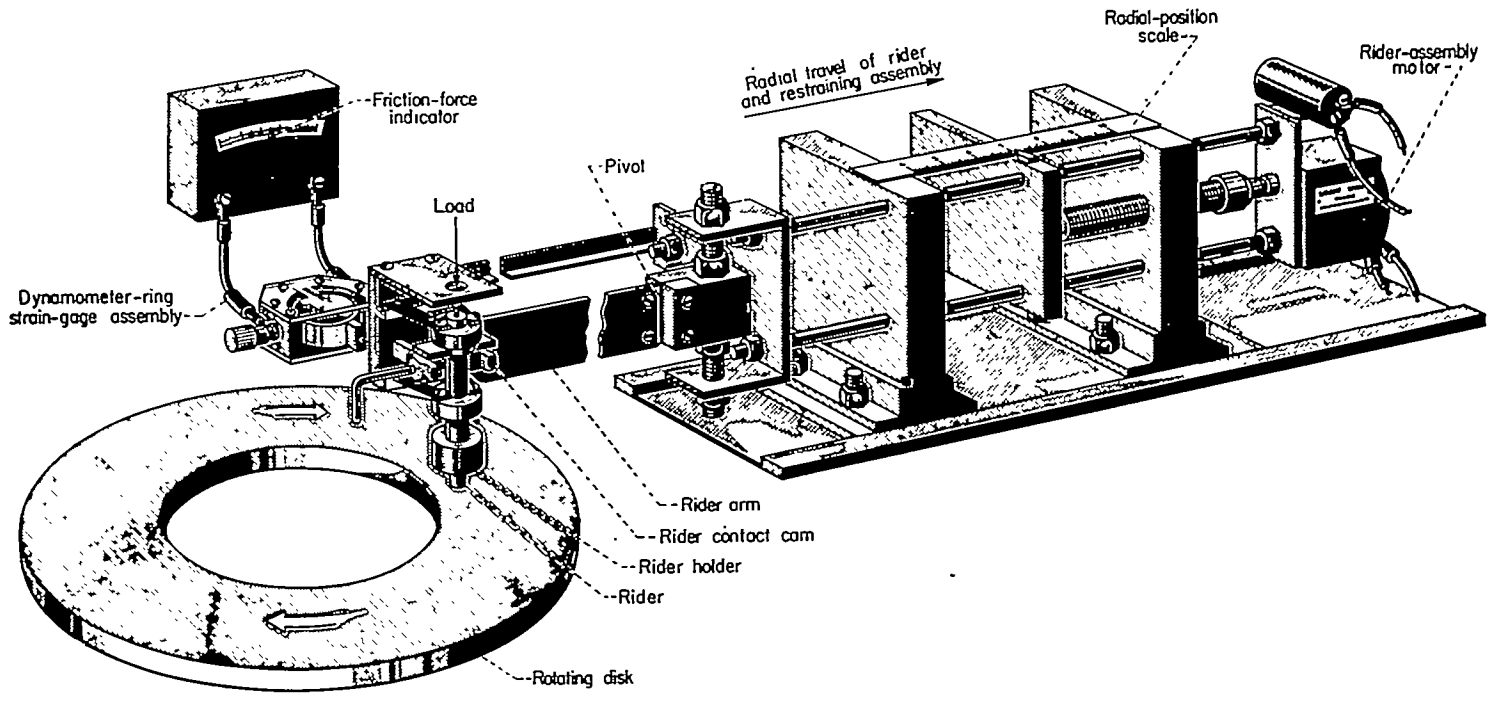



FIGURE 1.—Kinetic-friction apparatus; apparatus A (ref. 11).

experimental friction and wear studies were conducted with the kinetic friction apparatus shown in figure 1. The principal elements of the apparatus are the specimens, which are an elastically restrained spherical rider (either ⅛- or 3/16-in. radius) and a rotating disk 13 inches in diameter. The rider is loaded by a weight applied along its vertical axis. Friction force between the rider and the disk is measured by four strain gages mounted on a beryllium-copper dynamometer ring. The coefficient of friction is computed by dividing the measured friction force by the applied normal load: $\mu=F/W$. A radial-feed mechanism, when operating, causes the rider to traverse a spiral track on the rotating disk; the rider then slides on virgin surface of the disk. Where solid surface films were investigated, they were usually applied to the disk specimen before testing.

**Apparatus B; for kinetic friction (ref. 12).**—In some of the endurance runs to check the wear or life of bonded films, an apparatus similar in principle to apparatus A was used. It differs from apparatus A in that no radial traverse takes place; the rider thus slides in the same wear track on the disk specimen at all times. It is a modification of the apparatus described in detail in reference 12. The spherical rider specimens have a radius of 3/16 inch and the disk specimens are 2½ inches in diameter.

**Apparatus C; for static friction (ref. 13).**—The principal elements of the static-friction apparatus are a rider assembly, in which three ¼-inch steel balls are rigidly clamped, and a stationary horizontal plate on which the rider assembly is supported by the balls. The applied load was distributed equally among the three balls, and the rider assembly was moved over the plate by application of a horizontal tugging force. Friction-force measurements were made with strain gages mounted on a beryllium-copper dynamometer ring between the tugging force and the rider assembly. Force was applied slowly, elastically extending the dynamometer ring until the force was great enough to overcome static friction and slip of the carriage occurred. Initial slip of the rider assembly was determined by observation through a telescope equipped with cross hairs. When motion was observed, an indicating mark was made on the time-force record of the potentiometer chart.

**Apparatus D; for reciprocating sliding (ref. 2).**—Some of the earlier studies of naturally forming surface films were made with a reciprocating slider mechanism. It applied normal load to two contacting surfaces, and slowly reciprocated one of the surfaces.

**Apparatus E; for kinetic friction (ref. 14).**—Experiments on solid lubricants, used in loose powder form, were conducted on an apparatus similar in principle to apparatus A. A ring with three equally spaced axial nodes was used as the rider specimen. The nodes were ground with a cylindrical face of 2-inch radius, parallel to the ring radius, so that in theory, line contact existed between each of the three sliding nodes and the disk. The rider specimens were of SAE 4620 steel, case hardened to Rockwell C–62. The disk specimens were of SAE 1020 steel.

### MATERIALS AND PROCEDURE

Most of the studies have been made with both specimens of steel, with the films under study applied to the disk

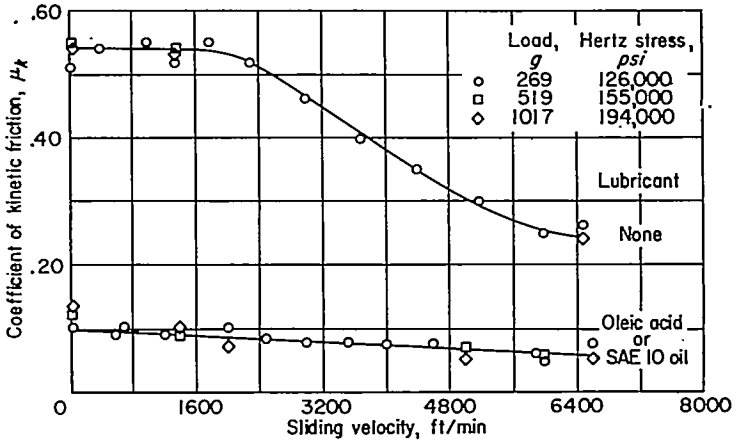



FIGURE 2.—Friction at high sliding velocities of steel on steel (ref. 11). Apparatus A; radius of spherical rider specimen, ⅛ inch.

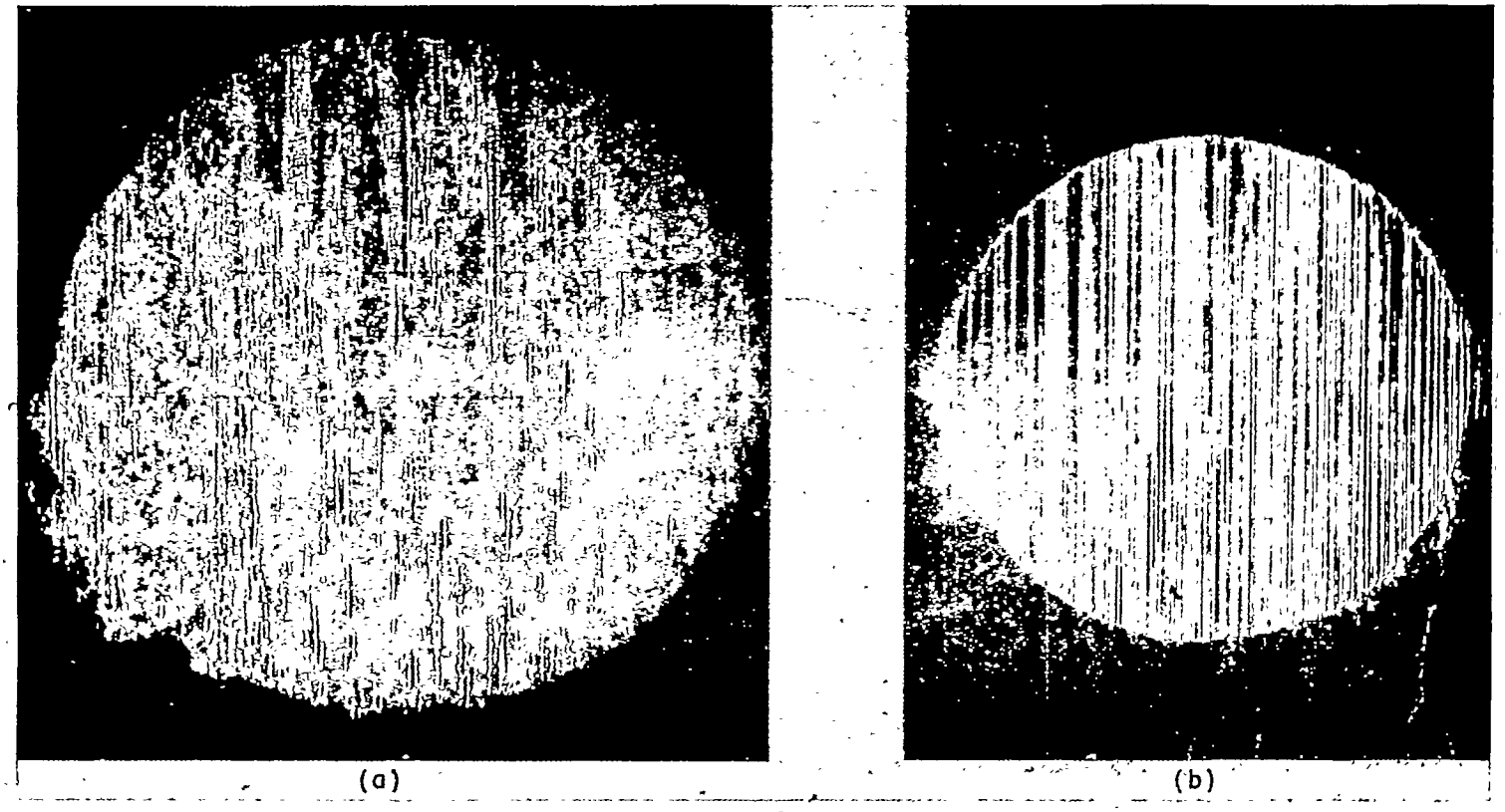
specimen. Some studies were made with a nonferrous alloy, such as one of the various nickel or copper alloys, as the spherically tipped (3/16-in. radius) rider specimen. Solid films investigated included various oxides, sulfides, and chlorides of iron, as well as materials such as molybdenum disulfide and graphite. Formation of various preformed films is described in detail in appendix B.

In most cases, the specimens were a sphere and a flat surface so as to enable calculation of both initial contact area and initial contact stress by the Hertz equations. The friction data presented are typical of data obtained in many runs. The limits of experimental error in the friction values presented were not uniform among all experiments because of the difficulties in maintaining absolute control of film thickness. In all but isolated cases, however, the maximum experimental error in friction coefficient, based on reproducibility, was $\pm 0.03$. In most cases, it was considerably less than 0.03. For comparative purposes, a load of 269 grams was used in obtaining most of the data presented. This load produces an initial Hertz surface stress (126,000 psi) that is in the range of contact stresses commonly present in aircraft engine components that require lubrication. According to reference 15, this stress is within the range of normal stresses (69,000 to 282,000 psi) for turbine-engine rolling contact bearings. At the same time, surfaces in contact under relatively light load and having a large apparent area of contact can have high stresses at localized contact areas (the contacting asperities). Even with lightly loaded surfaces, local pressure at these small points of contact is equal to the flow pressure of the materials (ref. 8) and causes plastic flow at these points.

## RESULTS AND DISCUSSION

### CLEAN STEEL ON CLEAN STEEL

For steel-on-steel surfaces cleaned by outgassing at 1000° C in vacuum, friction coefficients in vacuum as high as 3.5 have been measured (ref. 16). These surfaces were considered to be free of ordinary contaminants and most of the oxides. For steel-on-steel surfaces cleaned in air by the method presented in appendix B, maximum friction coefficient in air is 0.54 (fig. 2). The surfaces operating with a friction coefficient of 0.54 were known to have a film of $Fe_3O_4$ approxi-




(a) Clean and dry. (b) Effective boundary lubrication.
FIGURE 3.—Surfaces of rider specimens after tests of figure 2.

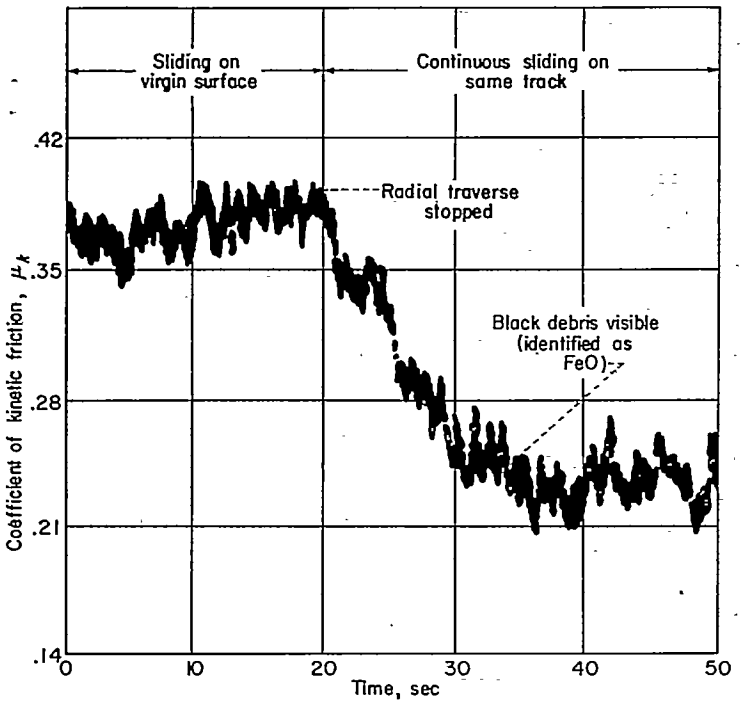



FIGURE 4.—Recording potentiometer tracing showing effect of high-velocity sliding over a continuous path (without radial traverse) on coefficient of kinetic friction. Black wear debris (ferrous oxide FeO) was visible at beginning of lower stable friction value (ref. 11). Apparatus A; unlubricated steel; load, 269 grams; sliding velocity, 4000 feet per minute; radius of spherical rider specimen, ⅛ inch.

mately 25 A thick (about $10^{-7}$ in.) (refs. 17 and 18). As sliding velocity is increased, friction coefficient is relatively constant at a value of 0.54 up to about 1600 feet per minute; above this value, a downward trend can be observed. For steel-on-steel surfaces lubricated with either oleic acid or SAE 10 lubricant, friction coefficient was approximately 0.10 at minimum sliding velocity, decreasing to 0.06 at 6600 feet per minute (fig. 2). For these results (discussed in more detail in ref. 11) the classical laws of friction are confirmed: friction coefficient is essentially independent of load and, at the lower speeds, is independent of sliding velocity.

Surface appearance of the ball specimens from the dry and the lubricated runs of figure 2 is shown in figure 3. The photomicrographs show that surface failure by welding occurred with the dry specimen; no surface failure (welding) is evident for the lubricated specimens but ploughing is apparent. The welding for dry steel was extensive, resulting in appreciable "tearing-out" and metal transfer.

### NATURALLY FORMED SURFACE FILMS

**Iron oxide films.**—In figure 2, the downward trend of friction coefficient for the dry steel specimens at the higher sliding velocities is not considered a violation of one of the classical friction laws (friction is independent of velocity); rather, it is considered the result of other variables entering into the mechanism. The downward trend is considered primarily a result of formation of beneficial iron oxide films of appreciable thickness; formation of oxide films would be accelerated at higher sliding velocities because of higher rate of heat generation at the sliding sufaces resulting from the greater release of frictional energy. In partial confirmation of this concept, when the steel slider was permitted to traverse the same wear track on the disk (fig. 4), FeO was identified (by X-ray diffraction) as the chief constituent in

the wear debris; there was a coincident reduction in friction coefficient from 0.38 to 0.24. It is reasonable to assume that the FeO film was being formed at all times; during the 30-second traverse of the same track on the disk, the film attained a thickness such that it became visible to the unaided eye. The gradual decrease in friction coefficient could be the result of the gradual increase in film thickness. Inasmuch as film thickness growth is limited by wear and rupture, friction coefficient would be expected to reach a stable value as film thickness stabilizes. No measurement of the film thickness could be made under the dynamic conditions; the appearance of the wear track was, however, somewhat similar to wear tracks produced on prepared oxide films approximately 1000 A thick.

It is hypothesized that, under sliding conditions of variable severity for specimens of steel on steel, surface oxides form in the order FeO to $Fe_3O_4$ to $Fe_2O_3$. Considerable confirming evidence (refs. 2, 4, 5, 16, 19, and 20) shows the importance of oxides in the reduction of friction, wear, and surface damage of sliding surfaces. On most run-in surfaces, oxides have been identified (refs. 2, 19, and 20). Figure 5, based on references 2 and 19 shows (qualitatively only) the estimated amounts of $Fe_3O_4$ and $Fe_2O_3$ present after various degrees of run-in. As shown, the amount of $Fe_3O_4$ increases to a maximum for the "fully run-in" condition; after further operation at higher load, less $Fe_3O_4$ is present. On the other hand, $Fe_2O_3$ shows a slight increase with degree of run-in to the "fully run-in" point, after which a marked increase in $Fe_2O_3$ is observed with failure of the surfaces. The surface films were identified by X-ray diffraction. In reference 19, it is shown that, at the minimum wear rate (which is usually associated with attainment of a "well run-in" condition), the amount of $Fe_3O_4$ is large; at the maximum wear rate (implying incipient surface failure), the amount of $Fe_3O_4$ is lower and the amount of $Fe_2O_3$ is larger than at the minimum wear rate. These data may also show why there has been considerable controversy in published literature about whether oxides of iron are good or bad from considerations of friction and wear. This controversy was an incentive for the NACA to obtain fundamental information on the specific oxides of iron: $Fe_3O_4$ and $Fe_2O_3$.

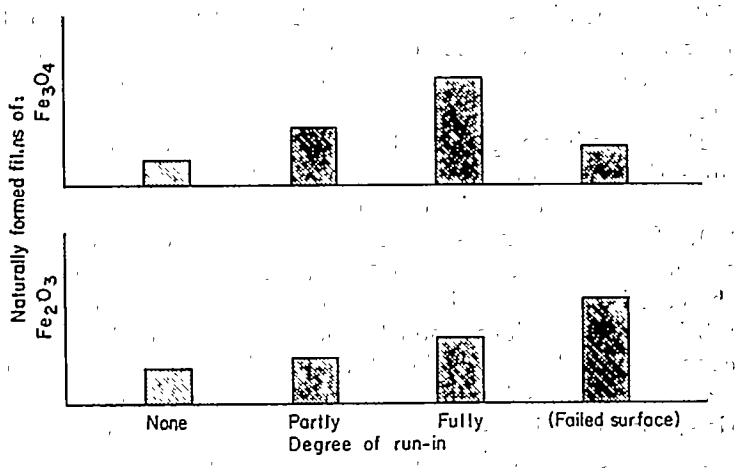



FIGURE 5.—Effect of run-in on formation of natural films of $Fe_3O_4$ and $Fe_2O_3$. Data are qualitative and are estimated from X-ray diffraction results of reference 2 (apparatus D) and from reference 19.

In agreement with the implications of figure 5, Finch (ref. 20) states that iron oxide layers are detected on most run-in surfaces. He indicates (as does Campbell, ref. 6) that oxides play a part in the mechanism of friction by affecting the ratio of welded to unwelded area. He also indicates that injurious oxides can be formed by excessive loading. This point is confirmed in the data of figure 5, which show that the oxide film $Fe_2O_3$ appears on the surface under "surface failure" conditions.

Recent experiments on the influence of adsorbed films were conducted by Bowden and Young (ref. 16). Their experiments showed the very marked effect of oxygen in reducing friction for iron-iron combinations that had been thoroughly outgassed at 1000° C in vacuum ($10^{-6}$ mm Hg). The surfaces used had a coefficient of friction of 0.4 before outgassing. Removal of contaminating films, including the oxides, increased coefficient of friction to 3.5, and led to complete seizure under certain conditions. Admission of oxygen to the vacuum chamber reduced friction coefficient from 3.5 to 1.2; with oxygen present, complete seizure no longer occurred.

Data of reference 21 show that exclusion of oxygen from clean specimens by use of a "blanketing" medium, such as highly purified cetane for steel-on-steel specimens, produces a high friction coefficient; these data are shown in figure 6. As sliding velocity increases above 2000 feet per minute, friction coefficient shows a downward trend for the dry specimens, but shows a very marked upward trend for similar specimens immersed in cetane, reaching a value greater than 1.0. The increase in friction is probably due to the absence of the beneficial oxide films; their formation would be prevented by the cetane, which excludes oxygen. Figure 6 shows that at low sliding velocities friction coefficient is lower for specimens with cetane than for dry specimens; the cetane may act as a contaminant between the surfaces. This beneficial effect might be expected to be greater at low than at high sliding velocities because of lower surface temperatures. Similar results on the action of benzene as a "blanketing" medium in metal cutting experiments are reported by Ernst and Merchant (ref. 22); they found that

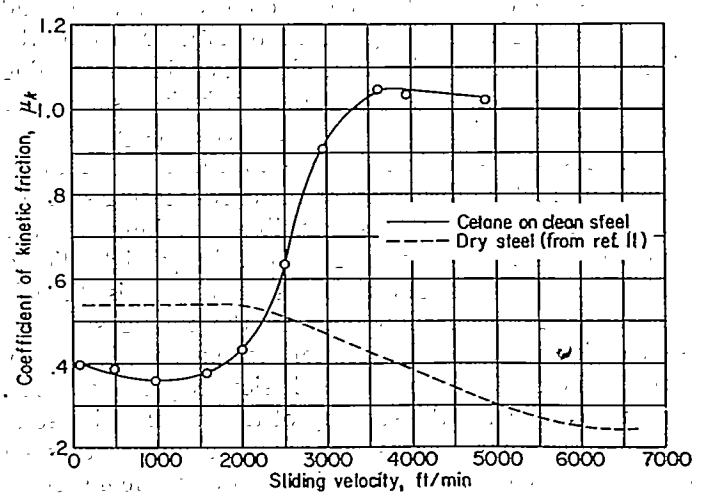



FIGURE 6.—Data showing effect of cetane as blanketing medium (to restrict availability of oxygen to specimen surfaces) on friction of steel against steel (ref. 21). Apparatus A; load, 269 grams; radius of spherical rider specimen, ⅛ inch.

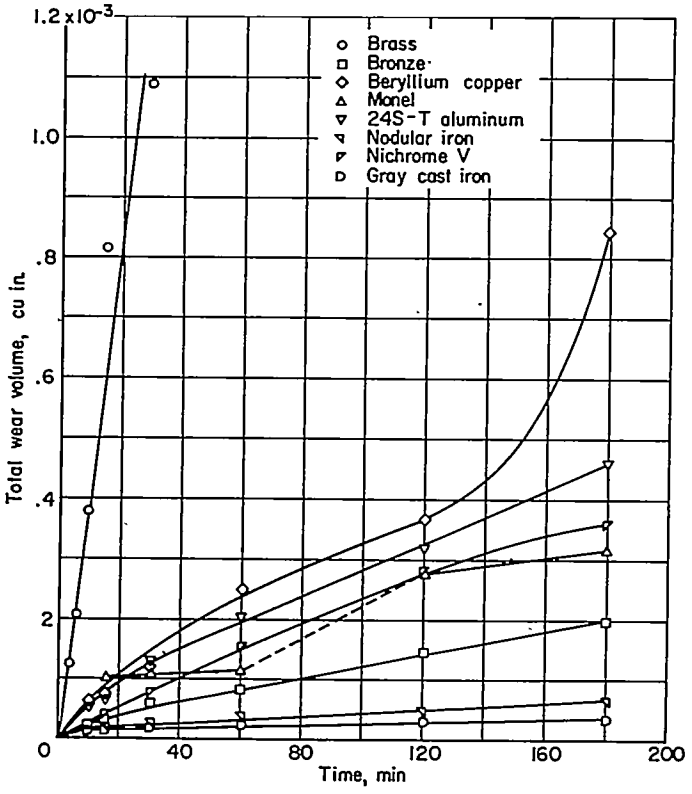



FIGURE 7.—Wear of several materials sliding against hardened SAE 52100 steel without lubricant (ref. 23). Apparatus A; sliding velocity, 5000 feet per minute; load, 50 grams; radius of spherical rider specimen, 3/16 inch.

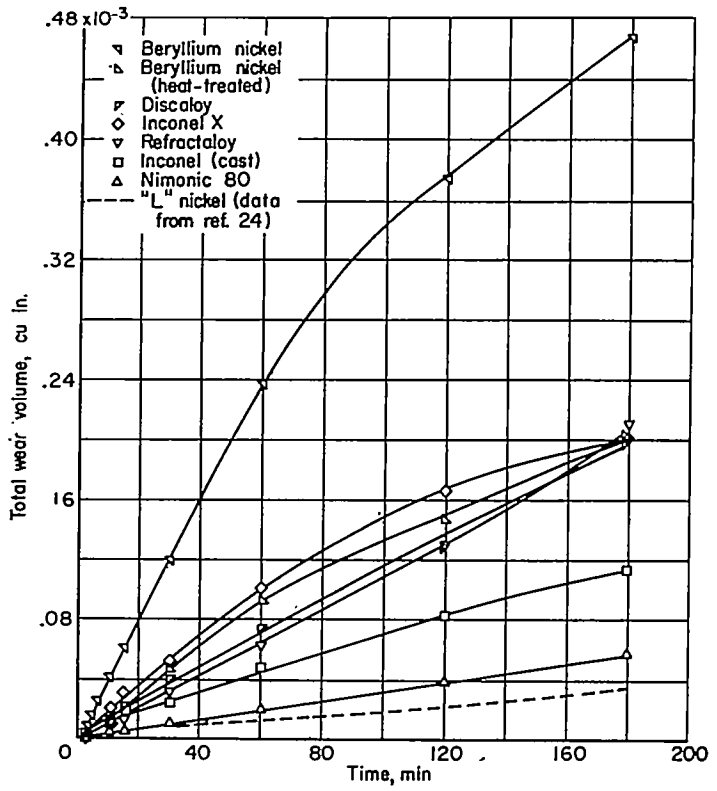



FIGURE 8.—Wear of several materials sliding against hardened SAE 52100 steel without lubricant (ref. 25). Apparatus A; sliding velocity, 5000 feet per minute; load, 50 grams; radius of spherical rider specimen, 3/16 inch.

friction coefficient was increased in the presence of benzene and decreased in its absence.

Thus, there is considerable evidence indicating the fundamental and important effect of oxides on the friction and surface damage of sliding metals. Not all oxides, however, are beneficial; detrimental oxide films can be produced under certain conditions (refs. 2 and 20). Additional discussion of the effect of specific oxides is given in the section Preformed Films.

**Miscellaneous films.**—In some practical applications, surface films have a marked effect on friction, wear, and damage of sliding surfaces (refs. 23 to 25). Most of the bearings employed in turbine-type aircraft engines are rolling contact bearings (ref. 23). One of the principal sources of failure in such bearings has been the cage (separator or retainer). As discussed in reference 23, most cage failures are caused by faulty lubrication at the cage-locating surface. This location is particularly susceptible to failure because the surfaces are in pure sliding at relatively high sliding velocities; also, the configuration is such that it is difficult to maintain a lubricant film between the sliding surfaces. These conditions reduce lubrication to extreme boundary conditions, and excessive metallic adhesion frequently occurs. One means of reducing the severity of this problem is use of a cage material that has less tendency to adhere to steel (under marginal conditions of lubrication) than the materials in current use. The NACA approach was to study the friction and wear properties of various materials, both dry and lubricated; the results were analyzed to determine the film-forming properties of the several materials. The studies of reference 1 show that the mechanism of lubrication prevailing in bearing materials containing low-shear-strength components and in cast iron containing graphite involves the smearing of the low-shear-strength phase over a hard matrix.

Results of the NACA study of cage materials are described in detail in reference 23. The investigation covered the sliding against SAE 52100 of materials such as bronze (containing 7.5 percent lead), beryllium copper, Nichrome V, nodular iron, and gray cast iron. Figure 7 shows some of the wear data obtained at a sliding velocity of 5000 feet per minute and a load of 50 grams. (For these experiments, the rider specimen, 3/16-in. radius, was of the various materials and the disk specimen was of SAE 52100 steel hardened to Rockwell C–60. Size of the wear spot on the rider was measured, and wear volume was calculated from this measurement.)

From this investigation, ability of materials to form surface films that prevent welding appears to be a most important factor in both dry friction and boundary lubrication (ref. 23). The surface films formed were derived from within the structure of the various materials (e. g., graphitic carbon in the cast iron and lead in the bronze). Under both dry and lubricated conditions, monel, Nichrome V, and beryllium copper formed films believed to be nickel oxides. When present, the films improved the performance of these materials. In this investigation, the films apparently had a greater effect on reduction of surface damage and of wear than on reduction of friction. The friction coefficient with the best materials of this investigation, the cast irons, was relatively high (approximately 0.5).

**Nickel oxide films.**—References 24 and 25 report an investigation of the wear and sliding friction properties of nickel alloys operated against various steels. Several monel metal alloys and other nickel alloys that have good high-temperature physical properties (such as Inconel and Nimonic 80) were investigated. As in the studies of reference 23, it was found that desirable performance characteristics and absence of extreme mass welding of all the materials studied could be associated with the development on the sliding surfaces of a naturally formed film. The film was identified as nickel oxide NiO by X-ray diffraction. Wear data for some of these alloys at a sliding velocity of 5000 feet per minute and a load of 50 grams are shown in figure 8 (note that the wear scale for figs. 7 and 8 are different). On the basis of wear and friction properties, cast Inconel performed very well in these experiments and compares favorably with nodular iron (which proved better than bronze, ref. 23). Nimonic 80 also showed promise as a possible cage material. Both of these nickel alloys have considerably better high-temperature properties than does nodular iron.

Other studies showing the important effect on wear of nickel oxide are described in the section Preformed Films.

Wear and friction experiments were conducted with some of the cage materials, listed in figures 7 and 8, sliding against disks of tool steels (molybdenum and tungsten types). The wear and friction results showed trends similar to those for tests against 52100 disks (figs. 7 and 8).

**Lacquer or varnish films.**—With reciprocating engines, good wear performance of piston ring—cylinder barrel combinations has been associated with formation of a very thin lacquer or varnish film on the surfaces. It has also been shown (ref. 26) that, for a journal bearing employing a lubricant of relatively poor lubricating ability or "film strength" (a silicone), formation of a lacquer film provides a margin of safety and increases the load-carrying capacity of the bearing.

The aircraft turbine engine is generally characterized by high operating temperatures; these high temperatures accelerate the process of oxidation, polymerization, and decomposition of the lubricant. These processes of degradation of lubricant result in formation of lacquer films on hot surfaces such as bearings. Because these films are naturally occurring, a fundamental evaluation of the role of such films in friction and surface damage would be worth while. Their influence on surface damage is particularly important in the case of the aircraft gas-turbine engine, which has a bearing or bearings operating dry for a short time after start of the engine. This condition is the result of the "high-temperature soak-back" of the turbine bearing after shutdown of the engine (refs. 27 and 28). Since the flow of coolant (lubricant) stops with shutdown, the bearing temperature increases because of the large reservoir of heat in adjacent large metal masses (such as the turbine wheel, immediately adjacent to the turbine bearing). Both Hunt (ref. 27) and Gurney (discussion, ref. 28) show that bearing temperatures on the order of 500° F prevail under the soak-back condition. When the engine is restarted after a period of shutdown, the bearings operate without lubricant for a short time until lubricant flow is established. During this initial period, the bearing parts (including the cage) are operating under conditions of dry friction. Because the cage has been a principal source of bearing failures and these cage failures have been established as lubrication failures (ref. 28), a study was made of the friction and surface-damage charactistics of films formed on steel surfaces by decomposition of several types of lubricant. These results are reported in the following section under **Lacquers or varnishes.**

### PREFORMED FILMS

Studies were made of a number of preformed solid surface films of the type formed: (1) naturally (as in the case of oxides), (2) by chemical reaction of surfaces with chemically active additives (extreme pressure lubricant additives), or (3) from solid lubricants (such as molybdenum disulfide and graphite) that function as supplemental lubricants (ref. 29). Formation of the various preformed films is described in detail in appendix B.

**Iron oxide films.**—Because of the importance of iron oxides, data were obtained on films (1200 A thick) of the specific oxides $Fe_3O_4$ and $Fe_2O_3$. These data, shown in figure 9, are from reference 30. The friction coefficients, as well as visual examination of the surfaces, show that $Fe_3O_4$ can be quite beneficial in decreasing friction and in preventing surface damage. In comparison, $Fe_2O_3$ showed high friction and excessive welding and surface damage. Figure 10 shows a comparison of the rider specimens after investigation; surface protection was much better with the $Fe_3O_4$, which largely prevented surface welding and metal transfer.

Confirming evidence of the importance of the specific iron oxide is available in references 31 and 32. Fretting (fretting corrosion) is a concentrated and severe form of wear. With specimens of steel against glass (ref. 31), the first wear particles produced during fretting were extremely small and apparently virgin material. These particles oxidized when exposed to air. Visual observations (ref. 31) showed color changes that suggested successive oxidation of

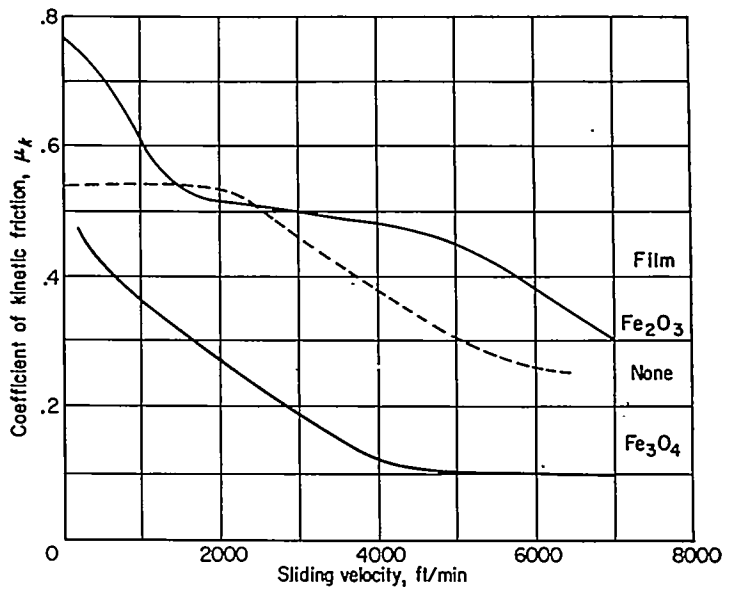
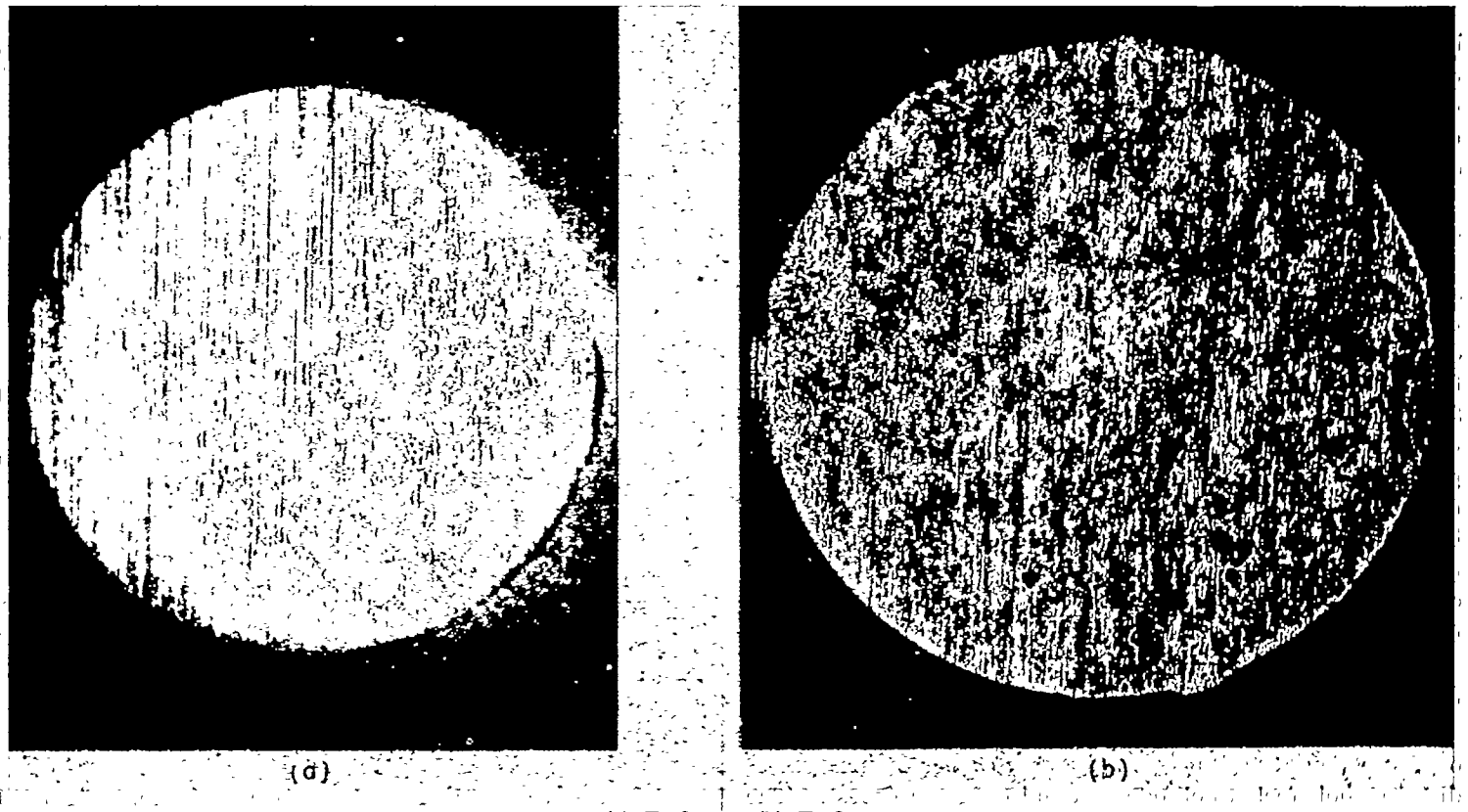



FIGURE 9.—Friction at high sliding velocities of dry unlubricated steel against steel with no film and with preformed films of $Fe_3O_4$ and $Fe_2O_3$ approximately 1200 A thick (ref. 30). Apparatus A; radius of spherical specimen, ⅛ inch.




(a) $Fe_3O_4$. (b) $Fe_2O_3$.

FIGURE 10.—Surfaces of rider specimens after tests of figure 9.

Fe to FeO, to $Fe_3O_4$, and finally to $Fe_2O_3$, which is hard and highly abrasive. Friction coefficient of powdered $Fe_2O_3$ compacts reciprocated against each other was relatively constant (after a few hundred cycles) at a value of approximately 0.5 (ref. 32). The friction coefficient of steel on steel (0.6) was not too different from that with the $Fe_2O_3$ compacts. With powdered $Fe_3O_4$ compacts reciprocating against each other, friction coefficient was at first low (0.3), but gradually increased until, at 600 cycles, the value was 0.5, the same as that for the $Fe_2O_3$ compacts. Examination and chemical analysis of the debris on the surface of the $Fe_3O_4$ compacts showed that $Fe_2O_3$ was present. Thus the fretting in all three cases, (1) steel on steel; (2) $Fe_2O_3$ on $Fe_2O_3$; and (3) $Fe_3O_4$ on $Fe_3O_4$, was, after a number of cycles, essentially that of $Fe_2O_3$ against $Fe_2O_3$ as suggested by the measured values of friction coefficient.

These data, as well as the data on naturally occurring iron oxide films previously discussed, emphasize the importance of iron oxide films to friction, wear, and surface damage. Effective lubrication under conditions of extreme boundary lubrication (where metal-to-metal contact takes place) is very often a function of the *nature* of the oxide films present. When surfaces are lubricated with fatty acids, oxide films may also contribute to the formation of metallic soap films at the surface. This point is developed in the section Metallic soaps.

**Chlorides and sulfides.**—The mechanism of the action of extreme pressure lubricants is considered to be one of chemical reaction between active additives and the metal surfaces. Compounds of chlorine, sulfur, or phosphorous are formed, depending upon the type of additive used.

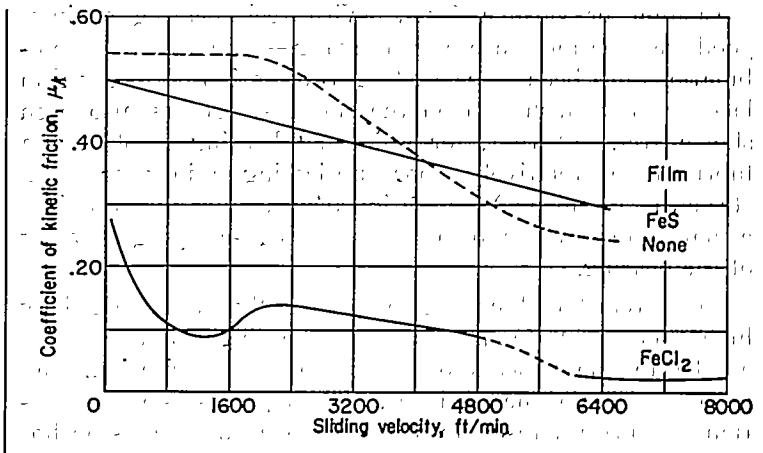



FIGURE 11.—Friction at high sliding velocities of steel against steel with no film and with preformed films of $FeCl_2$ and FeS approximately 1000 A thick (ref. 30). Apparatus A; radius of spherical rider specimen, ⅛ inch.

Results of an investigation (ref. 30) on preformed chloride and sulfide films approximately 1000 A thick are shown in figure 11. Visual examination of the surfaces showed that both FeS and $FeCl_2$ are effective in preventing excessive surface damage; $FeCl_2$ is much more effective than FeS in reducing friction. The difference in friction coefficient corresponds to the difference in shear strengths of the two compounds. If the reaction products from extreme-pressure lubricant additives of the chlorine and sulfur types are primarily $FeCl_2$ and FeS, respectively, the effectiveness of the additive containing chlorine should be appreciably superior. The superiority of chloride films over sulfide films

is confirmed by Bowden and Tabor (ref. 8), who found similar differences in friction coefficient.

Effectiveness of sulfide films in reducing friction has been shown to be very much a function of film thickness (refs. 6 and 13). Campbell shows that, on copper, friction coefficient decreases as sulfide film thickness increases up to about 1000 A. Results obtained by Greenhill (ref. 33) indicate that sulfide films on steel, copper, or silver showed maximum reduction in friction when the thickness exceeded about 1500 A. Under the conditions of experiments with sulfide films of FeS on steel reported in reference 13, a thickness of approximately 6000 A is required for minimum friction coefficient, although surface welding was effectively prevented by films 4500 A thick. These data are shown in figure 12. Since the data of figure 11 are for an FeS film of approximately 1000 A where welding was effectively prevented, the value of 4500 A for the studies of reference 13 appears to be peculiar to the conditions under which it was observed.

**Metallic soaps.**—There is rather general acceptance at the present time of the theory that boundary lubrication by materials such as fatty acids is effected primarily by chemisorption rather than physical adsorption. Lubrication is most effective, therefore, when there is some reaction of the fatty acids with the metals to form a metallic soap (refs. 8 and 34). Reference 8 shows that lubrication with fatty acids can be maintained at temperatures considerably above the melting temperature or desorption temperature of the fatty acids; in fact, the temperature at which lubrication failure takes place correlates remarkably well with the melting temperature of the metallic soap that is believed to be formed in the chemisorption of the fatty acids on the surface. It has been shown (ref. 8) that the formation of a metallic soap is very markedly affected by the presence or absence of oxide films. In experiments on steel, the results of reference 35 show that the *type* of oxide film is also extremely important. With a film of $Fe_2O_3$ on the surface, stearic acid in cetane was effective as a lubricant at sliding velocities only up to 3000 feet per minute; with a film of $Fe_3O_4$ on the surface, the same concentration of stearic acid in cetane was effective at sliding velocities higher than 7000 feet per minute.

**Solid lubricants.**—Because solid lubricants are frequently used as supplemental lubricants, they must be effective under conditions under which liquid lubricants become ineffective (ref. 29). In order to be effective, a solid lubricant must be

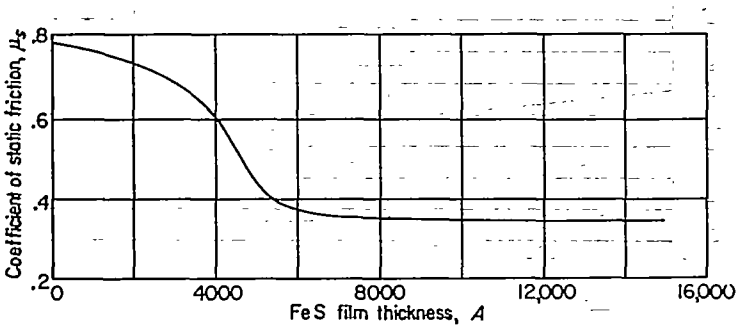



FIGURE 12.—Effect of thickness of film of FeS on static coefficient of friction of steel against steel (ref. 13). Apparatus C; load, 2400 grams; radius of spherical rider specimen, ⅛ inch.

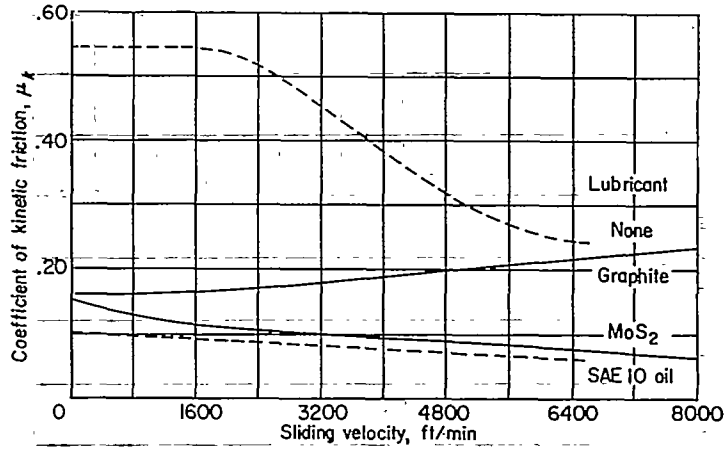


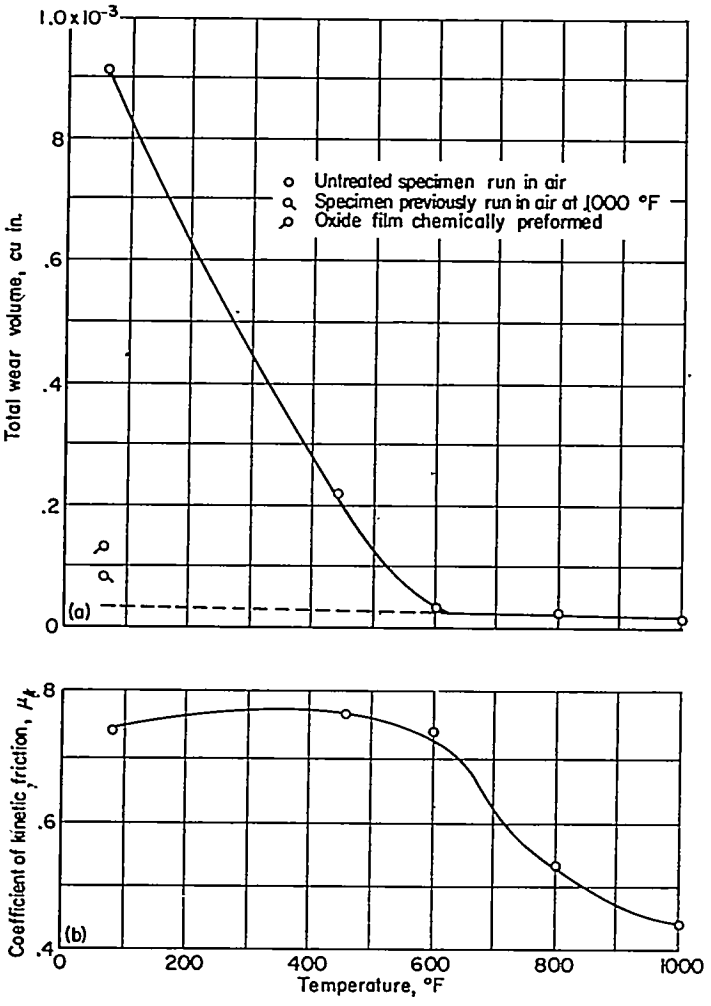

FIGURE 13.—Friction at high sliding velocities of steel against steel with preformed films of $MoS_2$ and graphite (ref. 30). Apparatus A; radius of spherical rider specimen, ⅛ inch.

maintained in place at the contact surface (ref. 36); that is, it must be strongly adherent to the surface and the film must be continuous. As indicated in reference 36, a "self-repairing" film is the most effective means of maintaining a film at the surface. Other types of film are, however, effective for limited periods of time.

In addition to the requirement that the lubricant film be strongly adherent to the surface, solids for use in the film must meet other requirements. Materials for this application should have the following physical and chemical properties: (1) high melting point, because of the high temperatures involved, (2) low hardness, because low hardness in a material is associated with low shear strength and consequently low friction (see appendix A), (3) laminar structure (some layer-lattice materials have been quite effective as solid lubricants), (4) limited solubility, (5) limited chemical reactivity, and (6) surface adherence. The five materials included in table I should be relatively effective in reducing friction and wear. On the basis of their properties, $MoS_2$ and graphite were indicated to be the materials of most interest.

Campbell (ref. 6) discussed the desired properties of solid lubricants and included, in addition to the mentioned points, the following: (1) The film should have good elastic properties; and (2) for a specific application, certain other properties may be required of the solids: high electrical conductivity, high thermal conductivity, corrosion inhibition, low density, small particle size, and freedom from abrasive contaminants. The requirement of freedom from abrasive contaminants is extremely important and is discussed later.

As shown in table I, both graphite and $MoS_2$ have excellent properties from the standpoint of possessing high melting point, laminar structure, low hardness (which corresponds to low shear strength), and relative chemical inertness. Since both of these materials have a laminar structure, shear within the material (in the plane of the laminae) should readily occur. Experimental friction results with $MoS_2$ and graphite are shown in figure 13; these data are discussed in detail in reference 30. At room temperature and low speed, there is little choice between the two materials. At the higher speeds, the friction coefficient of $MoS_2$ is lower. Visual observation of the rider specimens indicated that some




(a) Wear.
(b) Friction.

FIGURE 14.—Wear and friction of cast Inconel (rider) sliding against M–10 tool steel (disk) unlubricated. Apparatus B; sliding velocity 120 feet per minute; load, 1200 grams; radius of spherical rider specimen, 3/16 inch.

welding had occurred with the graphite film and none with the $MoS_2$ film. Occurrence of welding with the graphite film may have been affected by manner of film formation or by humidity. An important difference between the graphite and the $MoS_2$ films is the manner in which the two films were formed; the graphite film was a rubbed film and may not have adhered well or been completely continuous on the surface. The $MoS_2$ film was a continuous film, bonded to the surface by the corn syrup method described in appendix B. The second factor involves the presence or absence of adsorbed water films. Considerable research (conducted by Savage, Ramadanoff, Campbell, and others, and reviewed by Campbell, ref. 6) has shown that the presence of adsorbed water or oxygen films promotes ease of slip between adjacent crystal planes of graphite. Thus, in low humidities or in a vacuum, wear and friction with graphite are very high. Campbell also states that, if the graphite is properly run-in under normal conditions so that sufficient water is adsorbed, a high degree of orientation is produced with the slip layers parallel to the supporting surface. After such run-in, wear is very low even in relative humidities as low as 0.4 percent. With the NACA rubbed films, no run-in was attempted; thus, sliding under severe conditions (such as the higher sliding velocities of fig. 13) generated considerable frictional heat at the contacting surfaces. This heat may have desorbed any water films at the surface, and consequently reduced the lubricating effectiveness of the graphite.

**Nickel oxide.**—It was observed (refs. 24 and 25) that good performance characteristics (low wear and prevention of surface damage) of nickel alloys were obtained when a surface film of NiO was present; when no surface film was present, poor performance was obtained.

Research at this laboratory has shown that, with increase in temperature, wear of Inconel decreases. This result would be expected, since an oxide film could form more readily at the higher temperatures. The reduction in both wear and friction with increase in temperature is shown in figure 14. The data show that wear at temperatures between 600° and 1000° F is approximately one-twentieth that at 75° F. The effect of the film on wear was checked with two types of experiment: In the first, specimens with preformed NiO films were tested; in the second, an attempt was made to prevent the natural formation and repair of the oxide film by limiting the availability of oxygen to the specimens.

Films were preformed by two methods. One film was preformed on a cast Inconel specimen by making a wear run in air at 1000° F; a room-temperature wear run was then made with this same specimen. As indicated in figure 14 (a), the wear rate was approximately one-tenth of that obtained with an untreated specimen at room temperature. A film was preformed on a second cast Inconel specimen by heating it in molten caustic (NaOH). The specimen was then run at room temperature, and the data (fig. 14 (a)) show that wear was again approximately one-tenth of that obtained under similar conditions with an untreated specimen of the same material. The experiment involving oxygen availability was made with cast Inconel at 1000° F in an atmosphere of argon. Although some oxide was undoubtedly present (because air was present as a contaminant), wear was higher by a factor of 4. These results point to the beneficial effect of the nickel oxide film on both wear and prevention of surface damage.

**Lacquers or varnishes.**—The study on lacquers and varnishes (referred to in the section Naturally Formed Surface Films) was made on friction specimens to which had been applied lacquer or varnish films from decomposition of several

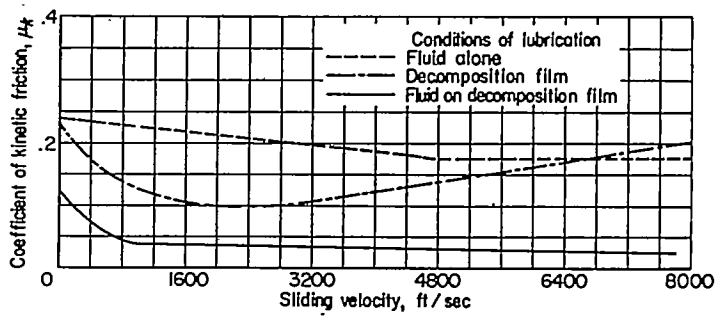



FIGURE 15.—Effect of sliding velocity on kinetic friction of steel against steel lubricated with silicone fluid and decomposition films formed from a silicone (ref. 37). Apparatus A; load, 269 grams; initial Hertz surface stress, 126,000 psi; radius of spherical rider specimen. 1/8 inch.

types of lubricant. These lubricant types included ordinary petroleums and synthetic lubricants of the diester, glycol, and silicone types. Results of this investigation are reported in detail in reference 37.

In general, the data of reference 37 show that the decomposition products reduced friction and surface damage of slider surfaces when compared with dry, clean steel surfaces. The effect of surface films in reducing friction and protecting the surfaces is strikingly demonstrated in figure 15, which shows friction coefficient under three conditions: (1) with silicone (a poor lubricant) alone, (2) with a silicone decomposition film, and (3) with silicone fluid over the decomposition film. The marked reduction in friction with decomposition films is apparent. Furthermore, with the fluid alone, surface damage was severe; appearance of the rider was similar to that shown in figure 3 (a). With the decomposition film alone and with the fluid over its decomposition film, damage to the surfaces was very slight; rider appearance was similar to that of figure 3 (b).

**Platings.**—Bowden and Tabor (ref. 8) have shown that plated films of soft metals can produce extremely low friction coefficients, provided the underlying metals do not contact each other through the film. This finding implies a limiting film thickness below which protection to the surfaces is inadequate. Bowden and Tabor found that the limiting thickness was of the order of $10^{-5}$ centimeter for indium films. Other metals such as lead, silver, cadmium, and copper have been used as friction-reducing platings. The data of reference 8 (pp. 113–114 and 95–97) confirm the theory (appendix A) that the lower the shear strength of the film material, the lower will be the value of friction. Their results showed that ". . . friction of copper>lead>indium and that the values of friction . . . are roughly proportional to the shear strengths of these metals." From friction results obtained with a steel slider sliding on surfaces of steel, copper, lead, and indium, Bowden and Tabor calculated shear strengths of these materials. These values are given in the following table (ref. 8, p. 97); measured shear strengths of the pure metals are included for comparison:

| Metal | Shear strength, g/mm² | |
|---|---|---|
| | Calculated from friction measurements | From direct measurement |
| Steel | 140,000 | 90,000 |
| Copper | 28,000 | 16,000 |
| Lead | 1,600 | 750 |
| Indium | 325 | 220 |

In a practical application involving the cages of rolling contact bearings for aircraft gas-turbine engines, silver has been used (ref. 38) as a plating material. Its use prevented excessive metal transfer and "pickup" of the bronze cage material to the steel bearing material during operation after high-temperature soak-back.

### OTHER METHODS OF LUBRICATION BY SOLIDS

In practice, solids may be used to lubricate by two methods: (1) inclusion of the solid as a minor constituent of the materials to be lubricated; this method provides lubrication by formation of a film on the surface from within the structure of the material, and (2) preformation of a surface film and constant repair of this film. In method (1), the bearing material can be made by powder metallurgy with a solid minor constituent that is an excellent lubricant. With method (2), the surfaces may have a preformed lubricant film, and some external means may be provided to replenish the material so as to maintain a continuous film.

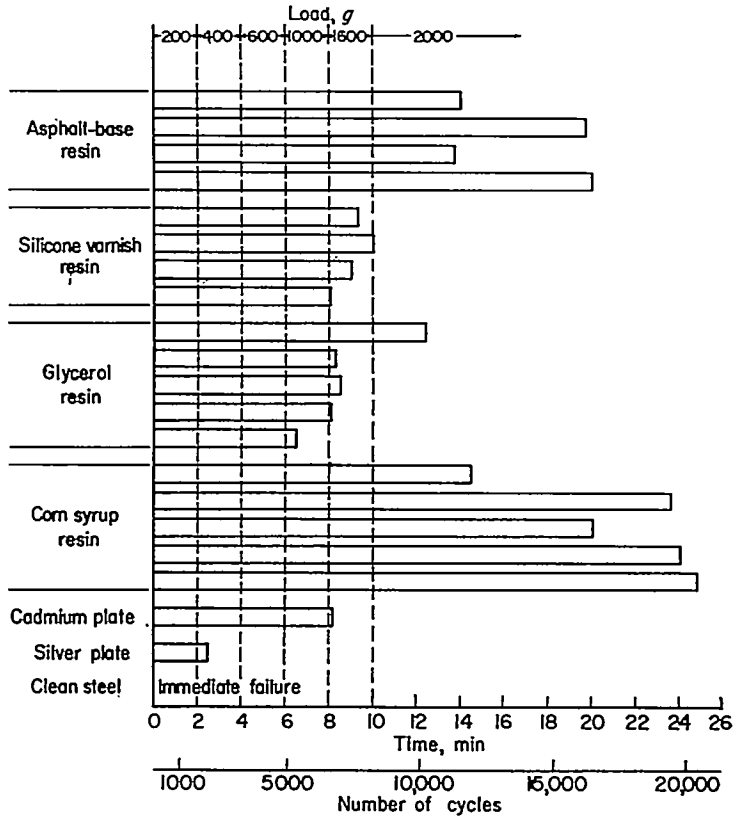



FIGURE 16.—Endurance tests showing total number of cycles to failure for several runs of solid-film lubricants of $MoS_2$ using four different resin-forming liquids. Cadmium-plated, silver-plated, and clean unlubricated specimens included for comparison. Film thicknesses, 0.0002 to 0.0005 inch. Failure was determined by high friction force and chattering of specimens (ref. 40). Apparatus B; radius of spherical specimens, ⅛ inch.

**Bonding of solid lubricants.**—References 39 and 40 cover investigations made to determine the mechanism of bonding of $MoS_2$ and other solid lubricants to various materials and to determine the friction and wear characteristics of such bonded films. It is shown in reference 39 that, when a solid powder suspended in a resin-forming liquid vehicle is applied to a hot surface, the liquid decomposes or polymerizes to resins that bind the particles of powder together and to the surface. Powders can be bonded in this way to materials such as steel, aluminum, brass, stainless steel, or glass. The resin-forming liquids that were investigated are: asphalt-base varnish, silicones, glycerin, ethylene glycol, polyglycol ether, and corn syrup. Solid lubricants bonded by this method were $MoS_2$, graphite, and $Fe_3O_4$. Electron diffraction studies (ref. 39) of a $MoS_2$ film bonded to steel showed no evidence of chemical reaction between the $MoS_2$ and the steel. As a part of the bonding investigation, rubbed films were also studied. Rubbing of $MoS_2$ and graphite was shown (refs. 39 and 30) to produce orientation of the materials.

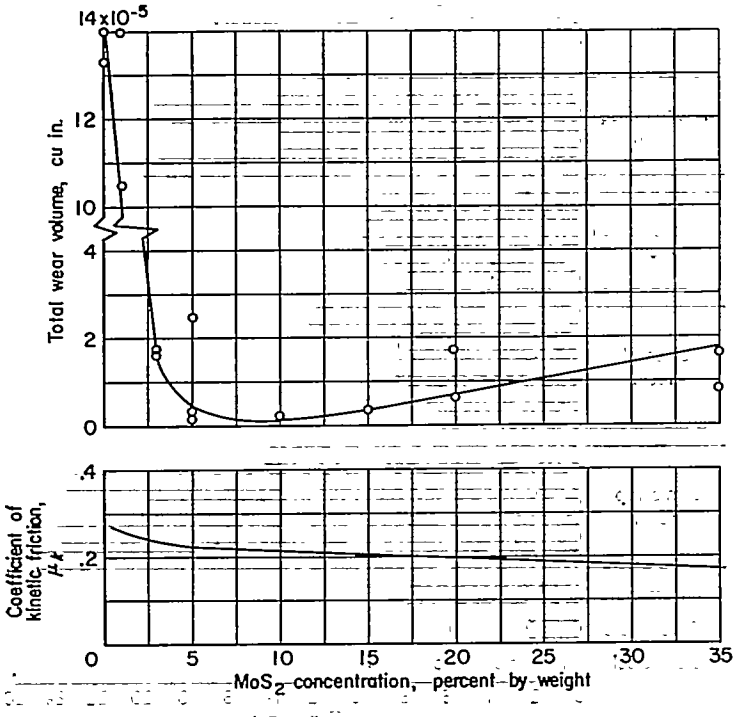



FIGURE 17.—Wear and friction of hot-pressed bearing material containing $MoS_2$. Rider specimens (on which wear was measured) were composed of 5 percent copper (by weight), 95 to 60 percent silver, and 0 to 35 percent $MoS_2$. Disk specimens were 1020 steel (ref. 41). Apparatus A; sliding velocity, 5000 feet per minute; load, 519 grams; time, 60 minutes; radius of spherical specimen, 3/16 inch.

The studies of reference 39 showed that resin-forming fluids on steel surfaces chemically reduce $Fe_2O_3$ to $Fe_3O_4$. This conclusion was reached from electron-diffraction studies of a $MoS_2$ film bonded with corn syrup as well as from chemical analyses of various mixtures of $MoS_2$, corn syrup, iron, and $Fe_2O_3$. Presence in the bonded $MoS_2$ film of $Fe_3O_4$ rather than $Fe_2O_3$ (the oxide originally present on the surface before the $MoS_2$ film was bonded) should be advantageous because of the better friction and surface protecting properties of $Fe_3O_4$ (fig. 9 and ref. 30).

**$MoS_2$ as a solid lubricant.**—The studies of reference 40 showed that solid lubricants can be applied by a practical bonding method. The reported friction and endurance data, obtained under the severe conditions of completely dry operation and high surface stresses, showed that solid-film lubricants (from 0.0002 to 0.0005 in. thick) of $MoS_2$ bonded with various resins (including corn syrup) possessed good lubricating qualities (fig. 16). For comparison purposes, figure 16 presents results for clean steel specimens and for specimens with a 0.0005-inch plating of cadmium or of silver.

An investigation was made (ref. 41) to study the lubricating effectiveness of $MoS_2$ included as a minor constituent in materials made by powder-metallurgy techniques; such bearing materials might be capable of operating successfully even under extreme boundary-lubrication conditions (possibly even completely dry). For these experiments, the rider specimens were powder-metallurgy compacts of: copper, 5 percent (by weight); silver, 95 to 60 percent; and $MoS_2$, 0 to 35 percent. Values of friction and wear of these materials are shown in figure 17. Wear was determined

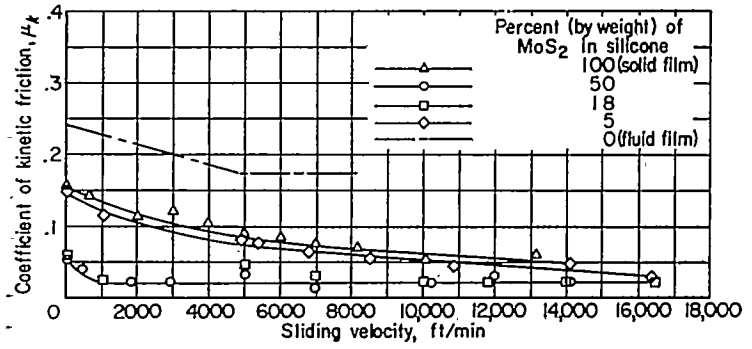



FIGURE 18.—Effect of sliding velocity on coefficient of friction for various mixtures of $MoS_2$ and a silicone fluid (a poor boundary lubricant) (ref. 43). Apparatus A; load, 269 grams; radius of spherical rider, 1/8 inch. Solid film of $MoS_2$ formed by corn syrup method described in appendix B.

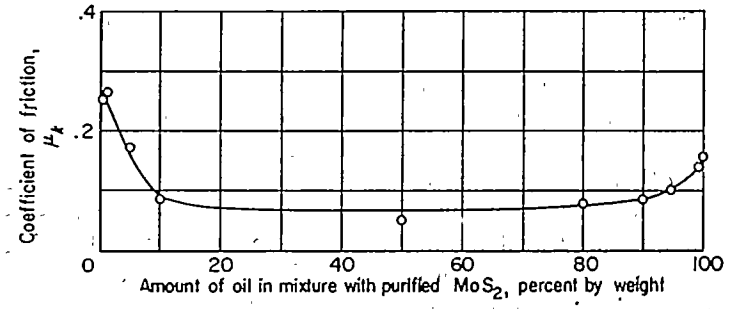



FIGURE 19.—Effect of mixtures of oil (white, medicinal) and $MoS_2$ on friction coefficient (ref. 44). Apparatus E; load, 40 pounds; sliding velocity, 6 feet per minute; radius of cylindrical rider specimen, 2 inches; steel on steel.

from the measured wear spot on the rider specimens. These data show that friction coefficient decreased progressively with increase in concentration of $MoS_2$. With wear, however, there was a definite optimum in $MoS_2$ concentration. High wear at low concentrations probably resulted from lack of effective lubrication; high wear at high concentrations probably resulted from lack of physical strength of the material. Since the specimens were operated without external lubrication, such materials show some promise for severe conditions of load, speed, and lack of lubrication. In these experiments, welding (as observed visually) was absent for all compacts that contained more than 5 percent $MoS_2$. The materials apparently formed an effective lubricating film on the surface by a transfer of solid lubricant from within the structure of the materials.

An investigation of lubrication by preformation of a surface film and constant repair of this film is reported in reference 42. The solid lubricant was supplied to the specimens with an "air-mist" system; the same effect could undoubtedly have been produced if the solid were supplied in a volatile liquid carrier. Preliminary investigations had shown that, with introduction of powdered $MoS_2$ in a rolling contact bearing, a continuous film of $MoS_2$ was formed on the contacting surfaces by the rolling action of the bearing components. It is possible (ref. 42) to operate conventional rolling contact bearings so lubricated either at high temperatures (approaching 1000° F) or at high speed (to $DN$ values of 900,000, equivalent to a rotative speed of approximately 12,000 rpm for a 75-mm-bore bearing).

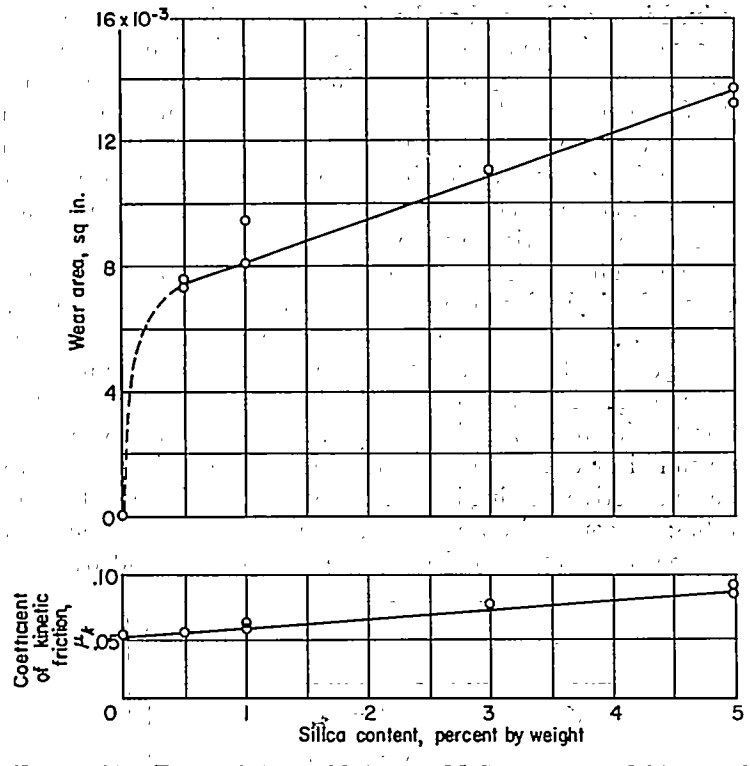



FIGURE 20.—Effect of silica additions to $MoS_2$ on wear and friction of steel specimens (ref. 44). Apparatus E; load, 40 pounds, sliding velocity, 6 feet per minute; duration, 6 hours; radius of cylindrical rider specimen, 2 inches; steel on steel.

Mixtures or suspensions of solids in liquid carriers may not at first consideration seem to fall in the class of solid surface-film lubricants as discussed in this report. Material so suspended may, however, provide lubrication by forming a solid film at the sliding surfaces. The studies presented in appendix E of reference 43 showed that low friction and complete protection of the surfaces from welding (as observed visually) could be obtained with mixtures of $MoS_2$ in a silicone fluid. These results were obtained with concentrations of 18 and 50 percent $MoS_2$ (fig. 18). The silicone fluid by itself was an extremely poor boundary lubricant for the steel-on-steel specimens of these experiments. Surface welding was prevented for $MoS_2$ concentrations as low as 5 percent. It should be recognized, however, that these particular mixtures were subject to settling, since they were not colloidal suspensions; such settling could be detrimental in a practical mechanism.

Mixtures of $MoS_2$ and a lubricant (white oil) produced friction coefficients (ref. 44) lower than those obtained with

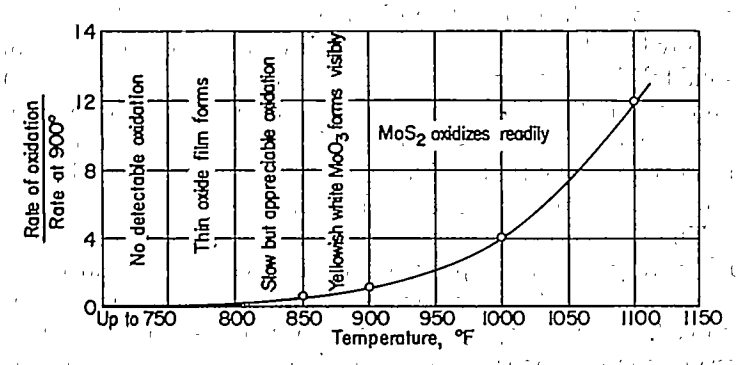



FIGURE 21.—Oxidation characteristics of $MoS_2$ in air. (Data from ref. 46.)

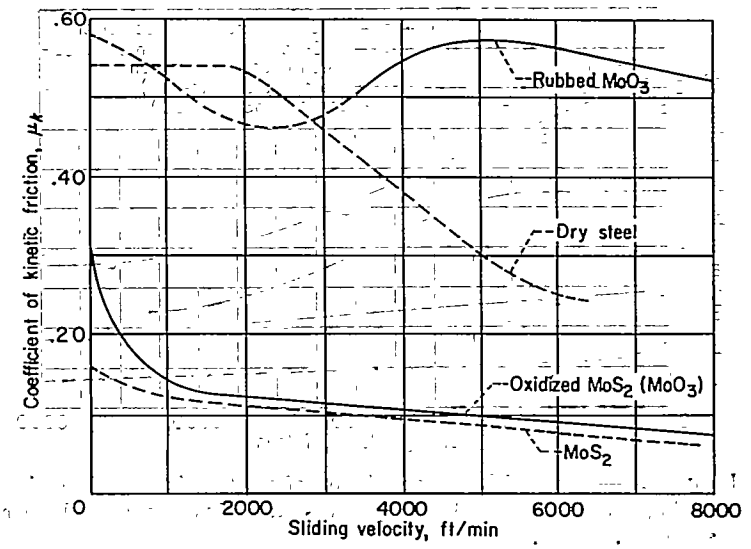



FIGURE 22.—Friction at high sliding velocities of steel against steel with preformed films of rubbed $MoO_3$ and oxidized $MoS_2$ (ref. 46). Apparatus A; radius of spherical rider specimen, ⅛ inch.

either the $MoS_2$ alone or the oil alone (fig. 19). The lowest concentration of $MoS_2$ required for minimum friction coefficient is approximately 10 percent.

As previously discussed, contaminants may be important in the use of solid lubricants. An investigation of these effects, using $MoS_2$ as the solid lubricant, is described in references 44 and 14. The studies of reference 44 revealed that naturally present contaminants, such as silica, might increase wear considerably, even though friction coefficient was increased only slightly (fig. 20). Moisture has a very detrimental effect on lubrication with $MoS_2$; both friction and wear increase as moisture content increases (ref. 14). Moisture in $MoS_2$ also forms acids that may be corrosive. Purity of the $MoS_2$ is therefore of considerable importance.

Campbell (ref. 6) states: "When chemical conversion coatings are used in combination with other solid lubricants, a surface is produced which outwears by a large factor either lubricant alone." His data show that marked increases in life (as measured by cycles to failure) could be obtained with a graphite film over a phosphated surface; the phosphate treatment was presumed to improve adherence of the lubricant film. Barwell and Milne (ref. 45) showed similar improvements (reduction in scuffing wear and increase in seizure load) by the use of $MoS_2$ in association with phosphated surfaces.

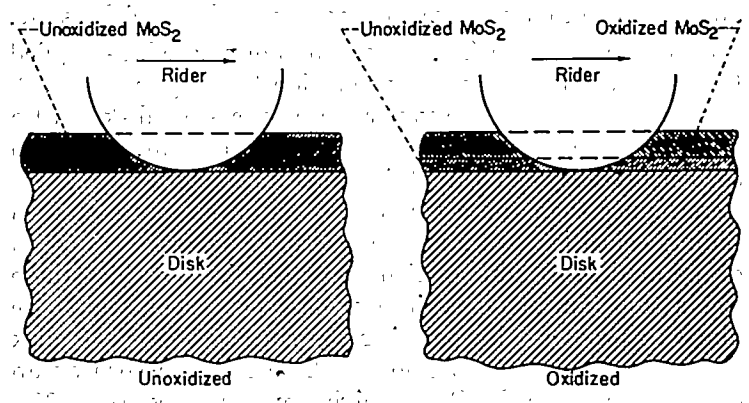



FIGURE 23.—Schematic representation of rider-disk combination, with unoxidized and with oxidized $MoS_2$ films.

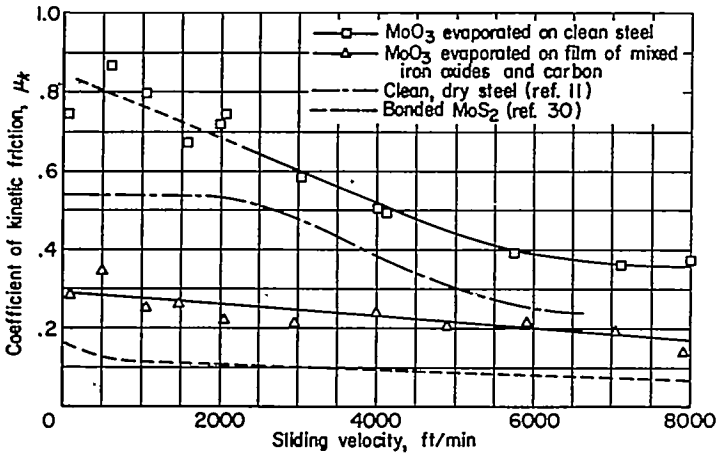



FIGURE 24.—Effect of $MoO_3$ on friction of steel against steel at high sliding velocities (ref. 46). Apparatus A; radius of spherical rider specimen, ⅛ inch.

Since $MoS_2$ appeared promising in the investigations at room temperature, it was considered as a high-temperature lubricant. An X-ray diffraction investigation of its chemical stability at high temperatures was accordingly made (ref. 46). This investigation showed that in vacuum there is no phase change of the $MoS_2$ at temperatures below 1000° F. In the presence of the oxygen, however, $MoS_2$ was found to oxidize to molybdenum trioxide $MoO_3$ at a very low rate at 750° F; the rate of oxidation increases steadily with increase in temperature as shown in figure 21. Since $MoO_3$ is abrasive, the friction characteristics of both an oxidized film of $MoS_2$ and a rubbed film of pure $MoO_3$ were studied. The results are shown in figure 22. The data show that $MoO_3$ is a very poor lubricant and its use resulted in high friction and excessive welding. The "oxidized" $MoS_2$ film, however, showed results remarkably similar to those for the unoxidized $MoS_2$ film.

An explanation of the mechanism of action of the $MoS_2$ in the "oxidized" condition must consider the actual surface films in both the oxidized and the unoxidized conditions. Sketches of the two surface films, based on an hypothesis that explains the mechanism, are shown in figure 23. While conditions in these experiments were intended to give as near complete oxidation as possible, undoubtedly some fraction of the $MoS_2$ remains unoxidized; even though this fraction is extremely small, it acts as an effective solid lubricant at the surface. The film immediately adjacent to the surface appears to be that part which produces the beneficial results.

Studies were made (ref. 46) of a film of pure $MoO_3$ applied to a clean steel disk by an evaporation technique; the $MoO_3$ was condensed from the vapor state on a steel disk until a film approximately 0.003 inch thick was present. Friction coefficients obtained with this film (upper curve of fig. 24) were higher than those for clean steel on clean steel at all sliding velocities. Studies were also made (ref. 46) of an evaporated $MoO_3$ film applied to a steel disk on which there was a thin film of mixed iron oxides and carbon; this base film was produced by painting the surface of the hot disk with corn syrup. As previously discussed, the corn syrup reduces $Fe_2O_3$ to $Fe_3O_4$ at high temperatures (ref. 39). Friction with the evaporated $MoO_3$ film applied over the mixed iron oxides and carbon is shown in figure 24; the data show that friction coefficient with this film is higher than that for a bonded $MoS_2$ film, but lower than that with either $MoO_3$ on clean steel or for clean, dry steel. This result again illustrates the importance of iron oxides to the friction mechanism; the reduction in friction (as compared with either $MoO_3$ on clean steel or with clean steel) is probably the result of surface protection by the $Fe_3O_4$ film.

The studies of reference 31 suggest that fretting damage is caused basically by high adhesive forces; the adhesion theory of friction predicts that a solid surface film of low shear strength should be effective in the mitigation, or inhibition, of fretting. Because of the effectiveness of $MoS_2$ as a solid-film lubricant, it was checked (ref. 47) for its effectiveness as an inhibitor of fretting. Data obtained that show $MoS_2$ to be an effective fretting inhibitor are presented in the following table; for comparison purposes, data for clean specimens and for specimens lubricated with mineral oil are included:

| Specimens | Surface film | Cycles to start of fretting |
|---|---|---|
| Steel ball on glass flat | None | 1-30 |
| | Mineral oil | 1,500 |
| | $MoS_2$ dusted | 72,000 |
| | $MoS_2$ bonded | 28,000,000 |
| Steel flat on steel flat | None | 100 |
| | $MoS_2$ bonded | 10,000,000 |

These results show that a surface film (even mineral oil) delays the start of fretting; this effect is believed to result from reduction of the adhesive forces responsible for fretting. Of the various methods of applying $MoS_2$, bonding of the film was clearly superior, probably because of the strong adherence of the bonded film to the surface.

**Other solids as lubricants.**—As previously stated, graphite is a solid that has many of the properties desired of a solid lubricant for use at high temperatures. In consequence, graphite was included in a previously unreported investigation in which lubrication of bearings at high temperature was studied; method of application of the graphite was essentially the "air-mist" system of reference 42. The investigation showed that lubrication of a 20-millimeter-bore ball bearing operating at 2500 rpm with graphite was successful at temperatures to 1000° F. Good results were obtained with both dried and undried graphite. In contrast to the harmful (abrasive and corrosive) products from oxidation of $MoS_2$, the oxidation products from graphite are "harmless" (ref. 6). As indicated in reference 6, "graphite starts to oxidize to carbon dioxide at 350° C (662° F)."

Boyd and Robertson (ref. 48) investigated a number of solid lubricants at extremely high pressure (to 400,000 lb/sq in.) and found that $MoS_2$ and graphite were among the best of the various lubricants included in their investigation. It is also possible that oxides of several metals used in the powdered form might effectively lubricate.

There have been several practical instances at the Lewis laboratory that have proved the effectiveness of solid lubricants where extremely severe operating conditions are involved. For example, a large number of air-handling valves are required to operate in a corrosive atmosphere at ambient temperatures up to 600° F. An attempt to operate the

valves with a bearing combination of a relatively hard stainless steel journal and a hard stellite bearing resulted in bearing seizure. A bearing was designed that had an interrupted surface with the surface interruptions filled with a solid lubricant. Dry bearings of this type have operated satisfactorily over a period of time at temperatures up to 600° F.

## SUMMARY OF RESULTS

Under extreme boundary-lubrication conditions, where metal-to-metal contact takes place, the generally accepted friction theory (according either to Bowden and Tabor or to Merchant) predicts that friction and tendency to surface failure (by welding) of rubbing metals can be reduced relatively simply; this reduction can be accomplished by a reduction of the ratio $s/p$ where $s$ is shear strength of the softer of the two contacting materials and $p$ is flow (yield) pressure of the softer of the two contacting materials. The most practical means of reducing the ratio $s/p$ is to reduce the value of $s$. The use of thin low-shear-strength films on hard base materials results in a reduction of $s$ with negligible reduction of $p$. Thus any low-shear-strength material (for example, some oxides, sulfides, plated films, liquid lubricants, etc.) that acts as a contaminant between sliding surfaces should be effective in reducing friction and surface failure. Experimental investigations by the NACA produced the following results, which are consistent with this view:

Experiments with iron oxides showed that FeO and $Fe_3O_4$ are generally beneficial, while $Fe_2O_3$ is harmful. With steel specimens in sliding, exclusion of oxygen by use of a blanketing medium prevented the formation of the beneficial oxides and permitted extensive surface welding. In fact, the results of these and other investigations indicate that, with many metals and particularly ferrous alloys, effective lubrication is very often dependent on the presence of an oxide film that can serve either as a solid lubricant film itself or as a means of obtaining a metallic soap film (by reaction with fatty acids) which serves as the solid lubricant. The importance of oxides to the friction and wear processes has not always been fully appreciated.

Chemical reaction films such as FeS and $FeCl_2$ were effective in preventing surface welding; $FeCl_2$ was much more effective than FeS in reducing friction (for example, at 3000 ft/min sliding velocity, friction coefficient $\mu$ was approximately 0.13 for $FeCl_2$ and 0.40 for FeS). This difference in friction corresponds to the difference in shear strengths of the two compounds.

Wear studies showed that prevention of surface damage and maintenance of low wear could be associated with the formation of naturally occurring surface films on one or both of the sliding specimens; for cast irons, the surface film consisted of graphitic carbon and for various nickel alloys, the film consisted of the nickel oxide NiO. In one investigation with cast Inconel sliding against hardened M–10 tool steel at temperatures ranging from 75° to 1000° F, a very marked downward trend of wear with increase of temperature was observed. It is speculated that this downward trend was the result of formation of beneficial nickel oxide(s) at the higher temperature. In this same investigation, wear was reduced by a factor of 10 through pretreatment of the Inconel specimen to form a nickel oxide surface film. It was possible to increase wear by limiting formation of the oxide film. This result was obtained by conducting tests at 1000° F in which the availability of oxygen was restricted by displacing the air surrounding the specimens with argon; under these conditions, wear increased.

Investigations of solid surface films of the lacquer or varnish type (preformed from decomposition of several petroleum and synthetic lubricants) showed that such films reduced friction and surface damage considerably, compared with dry clean steel surfaces. Furthermore, when a fluid silicone film is present over its lacquer or varnish film, the reduction in both friction and surface damage is quite marked as compared with the silicone film alone.

Various solids were considered for use as solid-film lubricants; of those considered, $MoS_2$ and graphite seemed best for use under severe operating conditions such as high temperatures, high loads, or any of the various operating conditions leading to extreme boundary lubrications. Investigations of these solids under high contact pressures, high temperatures, or high sliding velocities indicated that, in general, beneficial results were obtained with each material in the lubrication of steel on steel. These materials were therefore studied extensively with respect to: (1) methods of bonding to surfaces, (2) lubrication of rolling contact bearings, and (3) for $MoS_2$, stability at high temperatures.

Studies of the mechanism of bonding showed that both $MoS_2$ and graphite could be bonded to most surfaces by the use of a resin-forming liquid vehicle. The films so bonded were quite effective in preventing damage and reducing friction over limited periods of time. The $MoS_2$ and graphite were used to lubricate rolling contact bearings at high temperature and provided effective lubrication up to 1000° F. This application required a solid lubricant film to be formed and maintained (repaired) by continual injection of small amounts (approximately 0.001 lb/min) of the solid lubricant.

X-ray diffraction studies of $MoS_2$ showed no phase changes below 1000° F in the absence of oxygen. Electron and X-ray diffraction experiments show that in the presence of oxygen $MoS_2$ oxidizes to molybdenum trioxide $MoO_3$. The rate of oxidation increases steadily with ncrease in temperature: it is low below 750° F and becomes high at temperatures above 1050° F. Presence of a very small amount of unoxidized $MoS_2$ in oxidized bonded $MoS_2$ films will, however, maintain low friction and provide good protection from surface damage.

The general results of these investigations show that the presence of certain low-shear-strength solid surface films can markedly reduce friction, wear, and surface damage of rubbing metals operating under conditions of extreme boundary lubrication (where metal-to-metal contact takes place).

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, *February 10, 1955*

# APPENDIX A

## ADHESION THEORY OF FRICTION

Bowden and Tabor (ref. 8)

Friction, $F=S+P=\text{Shear}+\text{Ploughing}$

$$F=As+A'p$$

$$A=\frac{\text{Load}}{\text{Flow pressure}}=\frac{W}{p}$$

$$\mu=\frac{\text{Friction}}{\text{Load}}=\frac{F}{W}=\frac{As}{W}=\frac{A'p}{W}$$

$$\mu=\frac{s}{p}+\frac{A'p}{W}$$

When the ploughing term is negligible,

$$\mu=\frac{s}{p}=\frac{\text{Shear strength}}{\text{Flow pressure}}$$

Merchant (ref. 10)

$$\mu=\frac{s}{H}+\text{Tan}\ \theta=\text{Shear}+\text{Roughness}$$

When the roughness term is negligible,

$$\mu=\frac{s}{H}=\frac{\text{Shear strength}}{\text{Pressure surface hardness}}$$

where

| | |
|---|---|
| $F$ | friction force |
| $S$ | shear force |
| $P$ | ploughing force |
| $A$ | shear area |
| $s$ | shear strength |
| $A'$ | ploughing area |
| $p$ | flow pressure (in certain cases equivalent to yield strength in compression) |
| $W$ | load |
| $\theta$ | average angle that planes of slip make with general plane of surface |
| $H$ | pressure surface hardness (in certain cases equivalent to yield strength in compression) |

# APPENDIX B

## FILM PREPARATION

The preparation of various preformed films is described in considerable detail in references 13, 30, 37, 40, and 46; a condensed version of the procedure for each specific film is included in this appendix.

The finishing and cleaning procedure summarized here is described in detail in reference 11. Each disk was subjected to a similar milling, grinding, and lapping procedure, which was followed by a cleaning procedure essentially as follows:

(1) Degrease in low-aromatic cleaning naphtha
(2) Abrade with 3/0 emery paper
(3) Wash with mixture of 50 percent benzene and 50 percent acetone
(4) Scrub with levigated alumina
(5) Rinse with water
(6) Rinse with 190 proof ethyl alcohol
(7) Dry in clean, warm air

### FORMATION OF $Fe_3O_4$ FILM (REF. 30)

A film of $Fe_3O_4$ was formed by heating steel in a restricted oxygen supply; the disk was heated to 375° C (707° F) in a metal bell jar in which the air pressure was 0.1 millimeter of mercury. After 30 minutes in the vacuum furnace, interference colors were obtained which indicated a thickness in the order of 1200 A. The steel was cooled to room temperature in the furnace under the same low pressure.

### FORMATION OF $Fe_2O_3$ FILM (REF. 30)

A film of the oxide $Fe_2O_3$, which contains the greatest amount of oxygen of the iron oxide films investigated, was prepared by heating a steel disk to 350° C (662° F) in clean, dry air at atmospheric pressure. The disk was removed from the source of heat when the interference colors on the surface indicated thickness about 1200 A.

### FORMATION OF $FeCl_2$ FILM (REF. 30)

A film of $FeCl_2$ was formed by exposing a warmed, clean steel disk to the hot vapors composed of an azeotropic solution of hydrochloric acid and water. The warm disk was placed in a hot gas chamber at 100° C (212° F), which was then partly evacuated. When a valve was opened at the inlet to the chamber, a vapor composed of 20 percent water and 80 percent hydrochloric acid could envelop the disk. A 2-minute exposure was sufficient to form a thin, white, uniform coating believed (on basis of interference colors) to be approximately 1000 A thick.

### FORMATION OF FeS FILM

**Kinetic friction experiments of figure 11 (ref. 30).**—A film of FeS was formed by exposing a heated disk to hydrogen sulfide $H_2S$ gas. The clean steel disk was placed in a vacuum furnace at room temperature and the air pressure reduced to 0.25 millimeter of mercury. Hydrogen sulfide was admitted and the pressure again reduced. This process was repeated until the chamber was purged of air. The disk was then heated to 350° C (662° F) and an additional amount of $H_2S$ was admitted. The film formed rapidly and the furnace was allowed to cool while the low pressure was maintained. The film thickness was believed (on basis of interference colors) to be approximately 1000 A.

**Static friction experiments of figure 12 (ref. 13).**—Films of FeS were formed on the rider specimens (balls) by heating in an atmosphere of $H_2S$. Thickness of the films was calcu-

lated from weight gain and was checked by means of the modified chemical spot test, as discussed in reference 13. All weight gain was assumed to be caused by the addition of sulfur, which was stoichiometrically combined with iron to form iron sulfide.

#### FORMATION OF $MoS_2$ FILM

**Corn syrup resin for film of figure 13 (ref. 30).**—Molybdenum disulfide $MoS_2$ powder was mixed into a smooth paste with an organic binder (commercial corn syrup). The mixture was painted on a steel disk, the temperature of which was 350° C (662° F). After the disk cooled, loosely adhering $MoS_2$ was scraped off with a straight edge until the film was thin (approximately 0.005 in.), tenacious, gray-black, and very smooth. The disk was finished by light abrasion with 3/0 emery paper, followed by washing in 190 proof ethyl alcohol.

**Corn syrup resin for film of figure 16 (ref. 40).**—Equal parts by weight of $MoS_2$ and corn syrup were mixed to a paste. The disk was preheated to 250° to 300° C (482° to 572° F) and the paste applied to the disk. After the disk had cooled and the excess was scraped off, the surface was rubbed with fine steel wool and burnished with soft clean cloth to form a film of thickness from 0.0002 to 0.0005 inch.

**Asphalt-base resin (ref. 40).**—A mixture of 1 part (by weight) $MoS_2$ and 2 parts (by weight) asphalt-base varnish thinned 60 percent (by volume) with naphtha was brushed on a clean steel disk; the film was then air dried at room temperature until tack free. This film will become sufficiently hard (equivalent to hardness obtained when cured for 3 hr at 150° C) if enough time at room temperature is allowed. The disk was then rubbed with steel wool and burnished with a soft clean cloth to form a film of thickness from 0.0002 to 0.0005 inch.

**Silicone varnish resin (ref. 40).**—A mixture of 1 part (by weight) $MoS_2$ and 2 parts (by weight) silicone varnish thinned 20 percent (by volume) with xylene was brushed on a clean steel disk and dried under infrared rays until firm. The film was cured by heating at 200° to 250° C (392° to 482° F) for 3 hours. The disk was then scrubbed with steel wool and burnished with soft clean cloth to form a film thickness from 0.0002 to 0.0005 inch.

**Glycerol resin (ref. 40).**—A mixture of 1 part (by weight) $MoS_2$ and 2 parts (by weight) glycerin preboiled (to about one-fifth its original volume) was rubbed on a clean steel disk. The film was then dried either by (a) heating to 250° to 300° C (482° to 572° F) and repeating application of mixture with rubbing until film covered surface completely and continued heating until dry, or (b) drying under infrared until film was firm, then cured by heating for 3 hours at 250° C (482° F). The disk was then scrubbed with fine steel wool and burnished with soft clean cloth to form a film of thickness from 0.0002 to 0.0005 inch.

#### FORMATION OF OXIDIZED $MoS_2$ FOR FILM OF FIGURE 22 (REF. 46)

A disk, on which had been formed a film of $MoS_2$ in accordance with the method of reference 30, was placed in a muffle furnace and heated ½ hour at 1000° F. This treatment produced a top layer primarily of $MoO_3$, with an underlying thin layer of mixed $MoS_2$, iron oxides, and carbon.

#### FORMATION OF $MoO_3$

**Rubbed $MoO_3$ for films of figure 22 (ref. 46).**—Coarse, crystalline $MoO_3$ was rubbed on the flat disk to produce the rubbed $MoO_3$ film. This rubbing essentially resulted in filling the microscopic valleys of the steel surfaces.

**Evaporated $MoO_3$ for film of figure 24 (ref. 46).**—The $MoO_3$ was evaporated onto the surface of two disks. One of these disks was clean; the second had a film of mixed iron oxides and carbon on the surface. The mixture of iron oxides and carbon was obtained by painting the surface of a hot steel disk with corn syrup only. Evaporation of $MoO_3$ was done by heating molybdic acid $H_2MoO_4$ in a crucible; during heating, the acid dehydrated and became $MoO_3$, which melted, evaporated, then condensed on the cool disk surface immediately above the crucible. The films were approximately 0.003 inch thick. Details of the film preparation are given in reference 46.

#### FORMATION OF GRAPHITE FILM (REF. 30)

Graphite was deposited on the surface of the steel disk by compressing flaky graphite into a cake, which was then pressed against the rotating disk. The resulting film, while not completely continuous, was estimated to be less than 0.0005 inch thick.

#### FORMATION OF NICKEL OXIDE

Nickel oxide films were preformed by two methods. In the first, the film was preformed on a cast Inconel specimen by running a sliding friction experiment in air at 1000° F; previous sliding friction experiments with nickel alloys had shown that NiO was formed on the surface (refs. 24 and 25). In the second method, the film was preformed by heating a cast Inconel specimen in molten caustic NaOH. Exact thickness of these nickel oxide films is unknown.

#### FORMATION OF LACQUERS OR VARNISHES (REF. 37)

The lacquer or varnish films were formed by heating the disk specimen in air. Before heating, a thin film of fluid lubricant was uniformly deposited on the disk surface; in most cases, the quantity of fluid was approximately 1 cubic centimeter and the fluid was uniformly applied as a fine mist. Decomposition of the fluid lubricant was accomplished by heating the disks to temperatures slightly higher than those at which the first visible vaporization of the lubricant occurred. The temperatures were maintained as long as 14 hours.

#### PLATINGS

**Cadmium plating.**—The cadmium plate was applied using a lead anode in complex cyanide of cadmium bath. The thickness of the plate was 0.0005 inch.

**Silver plating.**—The silver plate was applied using a silver anode in a silver cyanide bath with aerosol as a brightener. The thickness of the plate was 0.0005 inch.

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TABLE I.—PHYSICAL AND CHEMICAL PROPERTIES OF SOLID LUBRICANTS

| Material | Melting point, °C | Hardness, mho | Crystalline properties | | | Solubility | | Chemical reactivity |
|---|---|---|---|---|---|---|---|---|
| | | | Structure | Form | Orientation | In water | Other solvents | |
| Molybdenum disulfide $MoS_2$ | 1185 | 1.0–2.5 | Hexagonal | Laminar | Random | Insoluble | $H_2SO_4$, aq. reg. | Medium |
| Tungsten disulfide $WS_2$ | 1250 | Friable | Hexagonal | Laminar | None | Insoluble | $HNO_3$+HF | Medium |
| Graphite C | 3527 | 1.0–2.0 | Hexagonal | Laminar | Highly preferred | Insoluble | None | Low |
| Lead iodide $PbI_2$ | 402 | ---------- | Hexagonal | Laminar | None | Slightly | KI | Medium |
| Silver sulfate $Ag_2SO_4$ | 652 | ---------- | Orthorhombic | ---------- | None | Slightly | $HNO_3$, $H_2SO_4$. | Medium |