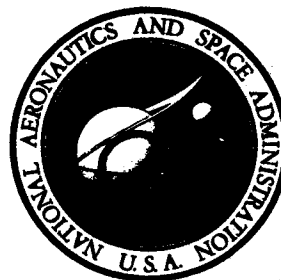


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**STUDIES OF LUBRICATING MATERIALS IN VACUUM**

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TECHNICAL PREPRINT prepared for United States  
Air Force - Southwest Research Institute Aerospace  
Bearing Conference  
San Antonio, Texas, March 25-27, 1964

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# STUDIES OF LUBRICATING MATERIALS IN VACUUM

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## SUMMARY

Lubricating materials for use in a vacuum environment have been the subject of a series of experimental investigations. Evaporation properties were evaluated for solid polymeric compositions. Friction and wear studies explored the behavior during sliding contact for series of polymeric compositions, binary alloys containing soft film-forming phases, complex alloys with film-forming materials, and a burnished MoS<sub>2</sub> film.

Friction and wear experiments were conducted at  $10^{-9}$  mm Hg with a 3/16-inch-radius-hemisphere rider specimen sliding on the flat surface of a rotating  $2\frac{1}{2}$ -inch-diameter disk specimen with materials that had low rates of evaporation. The influence of fillers in polytetrafluoroethylene (PTFE) on decomposition during vacuum friction studies was determined with a mass spectrometer. A real advantage in reducing decomposition and improving friction and wear properties is gained by adding fillers (e.g., copper) that improve thermal conductivity through the composite materials. A polyimide and an epoxy-MoS<sub>2</sub> composition material were found to have better friction and wear properties than PTFE compositions.

A series of alloys (cast binary as well as more complex alloys) that contained microinclusions of potential film-forming material was studied. These materials replaced the normal surface oxides as they were worn away on sliding contact. Iron sulfide, nickel oxide, and tin are typical film-forming materials employed and were demonstrated to be effective in inhibiting surface welding and reducing friction.

A burnished MoS<sub>2</sub> film applied to type 440-C stainless steel in argon with a rotating soft wire brush had good endurance properties but somewhat higher friction than commercially available bonded films. An oil film applied to the burnished MoS<sub>2</sub> markedly reduced its endurance life.

## INTRODUCTION

There are many types of mechanical devices that must be capable of a wide variety of duty cycles for extended time periods in space environment without the weight penalty of sealed systems (refs. 1 and 2). Conventional lubricants may not be useful for extended time periods because of evaporation or dissociation at low pressures (refs. 3 to 7). Conventional thin-film lubrication practice utilizes normal oxide films on metallic surfaces. When initial oxide films are worn away, ruptured, dissociated, or otherwise removed from bearing surfaces in oxygen-deficient environments such as space, conventional organic and other

lubricants cease to function in the usual manner (ref. 8). Further, when the lubricant and the normal protective oxide films are removed, most metals are subject to interface welding during sliding and rolling (refs. 9 to 11). Thus, low pressure and lack of oxygen are the two primary factors in space environment that make lubrication difficult.

The primary problem for lubrication of space devices is one of selecting designs and lubricating materials that will not suffer prohibitive evaporation or dissociation and will function to permit operation of surfaces in relative motion with low shear force (friction); mechanical parts should be made of materials having minimum susceptibility to surface welding. Special design consideration must be given to the difficult problems of dissipating frictional heat from devices operating in vacuum as well as of minimizing heat generation.

Based on experience from both laboratory experiments and bearing applications in satellite experiments, it has been found that two primary approaches to the problems of lubrication in space devices can be used. First, the systems concept provides for the operation of conventional lubricants and materials in sealed systems. Where low-vapor-pressure organic lubricants are used and the seal problem is in the regime of molecular flow with modest temperatures ( $<200^{\circ}$  F), significant endurance can be achieved for lightly loaded bearings. The radiometer bearings in Tiros II offer a good example of this systems concept (ref. 12). Because seal leakage is a function of pressure ratio in the regime of pressure flow, complex sealing assemblies are required where pressurized systems must be sealed against space environment for extended time periods.

Second, the materials concept requires the selection of stable materials for use in direct exposure to space environment. Frictional heat complicates the problem of degradation of lubricating materials. Films of inorganic compounds (e.g.,  $\text{MoS}_2$ ,  $\text{WS}_2$ ,  $\text{WSe}_2$ , etc.), soft metals (e.g., Ag, Au, etc.), solid polymers (e.g., polytetrafluoroethylene, polychlorotrifluoroethylene, polyimide, etc.), and liquid metals (e.g., gallium) have low vapor pressures and can provide lubrication for extended time periods in direct exposure to vacuum (refs. 1, 3, and 13 to 25). Any pre-formed film will have a finite endurance life that can be adversely affected by the evaporation tendencies and reduced heat transfer that are characteristic of vacuum operation. In regard to heat dissipation, the polymers require special consideration because of poor heat-transfer properties. In the interest of extended endurance lives, solid materials including a lubricating constituent in the structure can be useful (refs. 20 and 21). The use of filled polytetrafluoroethylene (PTFE) compositions, for example, for retainers of rolling-contact bearings, for ball sockets used in gimbal bearings, for idler gears to reform films on operating gears, and for sliding bearings can be useful.

Little has been done to develop optimum bearing materials and designs for vacuum operation. The performance of solid lubricants can be improved by proper selection of bearing materials (ref. 24). Design configurations

for minimum heat generation and optimum heat dissipation are desirable (ref. 1).

In the area of selection of bearing materials with least probability for harmful surface welding, it has been demonstrated that certain alloys, such as a cast cobalt alloy containing silicon, have useful properties (ref. 26). These properties may result from stable compounds present as inclusions in those alloys, but could also be a partial contribution of the hexagonal structure of cobalt. Compounds present as microinclusions in alloys replace the normal surface oxides as they are worn away. Almost any combination of clean metals will show surface-welding tendencies during sliding contact; both mechanical adhesion and alloying can be obtained at the interface when complete films of lubricants or other surface contaminants (such as oxides) cannot be maintained. Strong junctions can be established by (1) interface diffusion or alloying and (2) mechanical adhesion from interlocking of surface irregularities and plastic flow of materials. During relative motion, cold welding can occur even at cryogenic temperatures (ref. 27). Surface adhesion is a fundamental factor in lubrication processes according to the most widely accepted concepts of friction between solid surfaces (ref. 8). Metals with hexagonal crystal structures show less tendency for harmful adhesion than metals with cubic structures (ref. 28).

The objective of this paper is to review the recent research on lubricating materials for vacuum conducted at the NASA Lewis Research Center. The concepts on which this work was based have been previously reported and are summarized in the preceding paragraphs. The experimental evaluation of lubricating materials was obtained in friction and wear experiments performed in vacuum chambers at pressures of  $10^{-9}$  mm Hg or less. Evaporation experiments to determine stability of materials in vacuum have also been conducted. The types of materials investigated included solid polymers, solid inorganic lubricants, and cast alloys having film-forming microinclusions. The data reported herein are taken mostly from references 19, 20, 26, 29, 30, 31, and 32.

## APPARATUS

### Evaporation Studies

The evaporation-rate apparatus used in this study is shown schematically in figure 1. The specimen, which had a 5/16-inch-diameter exposed surface area, was suspended in a platinum pan from an electronic balance (sensitivity,  $\pm 0.01$  mg) into the mouth of a wire-wound tungsten cylindrical furnace. About 1.0 inch above the furnace was a copper condensing plate that was liquid-nitrogen cooled. The liquid-nitrogen-cooled shield was provided to condense the evaporating species as completely as possible. The entire apparatus was enclosed in a glass bell jar system, and pressure within the system was measured with a nude hot-cathode ionization gage adjacent to the evaporating system.

## Friction and Wear Studies

The apparatus used in this investigation is shown in figure 2. The basic elements of the apparatus were the specimens (a  $\frac{1}{2}$ -in.-diam. flat disk and a  $\frac{3}{16}$ -in.-rad. rider) mounted in a vacuum chamber. The disk specimen was driven by a magnetic drive coupling. The coupling had two 20-pole magnets 0.150 inch apart with a 0.030-inch diaphragm between magnet faces. The driver magnet that was outside the vacuum chamber was coupled to a hydraulic motor. The internal magnet was covered completely with a nickel-alloy housing (cutaway drawing in fig. 2) and was mounted on one end of the shaft within the chamber. The end of the shaft that was opposite the magnet carried the disk specimen.

The rider specimen was supported in the specimen chamber by an arm that was mounted from a gimbal and sealed to the chamber with a bellows. A linkage at the opposite end of the restraining arm from the rider specimen, was connected to a strain-gage assembly that was used to measure frictional force. The load was applied through a deadweight loading system.

Attached to the lower end of the specimen chamber was a 400-liter-per-second ionization pump and a mechanical forepump with zeolite and liquid-nitrogen cold traps. The pressure in the chamber was measured adjacent to the specimen with a nude hot-cathode ionization gage. In the same plane as the specimens and ionization gage was a diatron-type mass spectrometer for determination of gases present in the vacuum system. A coil made from 20 feet of  $\frac{3}{16}$ -inch-diameter stainless steel was used for liquid-nitrogen or liquid-helium cryopumping of the vacuum system.

## EXPERIMENTAL PROCEDURE

### Evaporation Studies

Each sample was placed into the system, and the system was then evacuated. When an ambient pressure of  $10^{-7}$  to  $10^{-8}$  mm Hg was reached, a 2-hour outgassing of the specimen at  $200^{\circ}$  was initiated. The purpose of the outgassing was to remove entrained gases and water from the specimens. In some instances (e.g., PTFE) outgassing periods to 24 hours were used, and the results were compared with those obtained for the shorter time period. After completion of the outgassing phase, the sample was cooled to room temperature, and the evaporation experiments were started. At the lower ambient temperatures, the specimen was held at specific temperatures for as long as 8 hours; at higher temperatures, meaningful data could be obtained in much shorter periods of time.

### Friction and Wear Studies

The specimens used in the friction and wear experiments were machined to size. The metal disks were circumferentially finish ground to a surface

roughness of 4 to 8 microinches. Before each experiment the metal specimens were given the same preparatory treatment: (1) thorough rinsing with acetone to remove oil and grease, (2) polishing with moist levigated alumina on a soft polishing cloth, and (3) thorough rinsing with tap water followed by ethyl alcohol. For each experiment a new set of specimens was used.

## RESULTS AND DISCUSSION

### Evaporation Studies

One of the important properties that must be considered in the selection of lubricating materials for space applications is evaporation tendencies. Evaporation-rate experiments were therefore conducted with various materials in vacuum.

The evaporation rates for three polytetrafluoroethylene (PTFE) compositions were determined. The materials examined were an extruded PTFE composition and two molded materials, PTFE No. 1 and PTFE No. 6. The results of figure 3 indicate that all three compositions exhibited lower evaporation rates at the higher temperatures than did an early commercial PTFE composition (shown as the reference curve in fig. 3). This may have resulted from such factors as improved purity (lower number of contaminants to initiate decomposition mechanisms), more efficient polymerization, and/or a narrower molecular weight distribution range (with higher average molecular weight). The extruded composition exhibited a higher evaporation rate than the two molded compositions. With both molded compositions a relatively low rate of evaporation was measured at ambient temperatures to 800° F. Above 800° F, as indicated from the data, decomposition began to influence markedly the measured evaporation rates. The results indicate that, if minimum evaporation rate is required, the molded compositions may be more desirable.

A relatively new development in solid polymers is the polyimide (ref. 31). In order to determine the stability of this material in vacuum some evaporation-rate experiments were conducted. The results obtained in these experiments are presented in figure 4. At ambient temperatures to 500° F the evaporation rate was less than  $10^{-10}$  gram per square centimeter per second. Above 500° F the weight loss was quite high since the material begins to decompose in this region. At temperatures to 500° F, however, the polyimide material appears very attractive for use in vacuum.

The evaporation rates for two epoxy compositions were also determined in vacuum. The material compositions were epoxy MoS<sub>2</sub> and epoxy PTFE. The results obtained are presented in figure 5. The evaporation rate for the epoxy PTFE composition was similar to that observed with the PTFE at temperatures below 700° F. Above 700° F the evaporation rate began to increase markedly. The epoxy MoS<sub>2</sub> composition exhibited no measurable evaporation rate at ambient temperatures to 600° F. Above 600° F, however, the weight loss increased appreciably.

Figure 6 is presented as a basis for comparing the evaporation data for the polymers just described with other lubricating materials of interest. The data shown for PTFE was for the early commercial product reported in reference 3. Important to the subsequent discussion are the relative evaporation rates for  $\text{MoS}_2$  and tin since they are utilized in friction and wear experiments to be described.

#### Stability of Oxides

The data of figure 7 indicate the oxygen partial pressures that are required for the dissociation of metal oxides at various temperatures. At all oxygen pressures above the curve, oxidation of metal can occur, while at all pressures below the curve, dissociation of the oxide occurs in accordance with the equation that accompanies the curve. As the temperature is increased, the reaction kinetics (rate) for the formation of surface oxides is increased. If the oxygen pressure is held constant, however, the tendency for the oxygen to bond to the metal will decrease, and at some point (curve) the energy is great enough to inhibit oxide formation. Examination of the data of figure 7 indicates that, at room temperature, in order to dissociate surface oxides and to prevent further oxide formation on iron, oxygen partial pressures of less than  $10^{-30}$  mm Hg are required. Figure 7 only indicates a concentration effect and not the kinetics of surface reactions at pressures above those that are necessary to achieve dissociation.

The stability of  $\text{NiO}$  is similar to that for the iron oxides. Thus if  $\text{NiO}$  can be utilized as a surface film it should not be subject to dissociation except at extreme temperatures. Metal sulfides and other inorganic compounds also have good stability in vacuum (e.g.,  $\text{MoS}_2$  in fig. 6).

#### Friction and Wear of Polymers

Friction and wear experiments were conducted in vacuum with PTFE and PCFE (polychlorotrifluoroethylene) compositions sliding on 440-C stainless steel (ref. 20). The results obtained are presented in figure 8. These results were obtained at a sliding velocity of 390 feet per minute with a load of 1000 grams on the polymer rider specimen, at a pressure of  $10^{-9}$  mm Hg and no external specimen heating. The friction coefficients obtained for all PTFE and PCFE compositions of figure 8 were between 0.25 and 0.30, which indicated that the presence of fillers in PTFE and PCFE had very little effect on friction coefficient.

The wear of the unfilled PTFE and PCFE were high and about the same value, which indicated that the presence of chlorine in the PCFE molecule exerted no influence on either friction or wear of PCFE. Large particles of the polymer abraded from the rider specimen surface adhered to the disk specimen surface. The sticky nature of the particles indicated that heat generated at the sliding interface was transferred to the polymer particles, which increased the surface temperature of the particles and caused localized or surface degradation. The polymer particles were thus absorbing frictional heat generated in the sliding process.



The addition of 25-percent glass fiber to PTFE did not influence markedly the friction obtained with PTFE; it did, however, decrease the rider wear by a factor of 100 (fig. 8). With the 25-percent glass-fiber-filled PCFE the specimen swelled and completely lost its shape (photographic insert of fig. 8) so that rider wear could not be measured. The softening point for this material is about 400° F. The swelling and presence of gas pockets within the specimen indicated that the specimen must have reached 400° F. This result was not obtained with glass-fiber-filled PTFE, which has a softening point of about 550° F. Examination of the wear area of the glass-filled PTFE composition revealed the presence of very fine black "sootlike" particles about the worn area. Chemical analysis indicated that the black debris contained iron, but about 70 percent was carbon. This film will be discussed later.

With the PCFE compositions, the fact that the unfilled composition did not swell and lose its shape as did the glass-filled composition may have resulted from the ability of the unfilled PCFE to dissipate a large quantity of heat in the wear particles. The glass-filled composition exhibited much less wear, and the heat generated at the sliding interface was therefore absorbed to a large extent by the rider specimen. The presence of glass fibers in the polymer may have increased the ability of the heat generated to be carried within the polymer body and to the rider specimen holder. (Thermal conductivity at 68° F in Btu/(hr)(sq ft)(°F)(ft) for PCFE is 0.145; for glass, 0.80.) Thus, the polymer temperature was not so high as it might have been without the dissipation of some heat to the metal specimen holder.

The friction and wear of a 25-percent-copper-powder-filled PTFE composite was also determined in vacuum. The friction coefficient obtained was nearly the same as obtained with the unfilled PTFE (fig. 8). The wear, however, was 100 times less and was comparable with that obtained with glass-filled PTFE.

Changing the sliding velocity will change the surface temperatures. In an attempt to gain some understanding of the polymer decomposition mechanism at the sliding interface, some friction experiments were conducted at various sliding velocities using the mass spectrometer to identify the decomposition products formed. A plot of the decomposition products formed (ion concentration) as a function of sliding velocity for various PTFE compositions is presented in figures 9 to 11.

In figure 9, with unfilled PTFE sliding on 440-C stainless steel, the principal decomposition products observed were mass-charge ratios M/e of 31 ( $\text{CF}^+$ ) and 50 ( $\text{CF}_2^+$ ). It is interesting to note that as the sliding velocity increased a decrease in ion concentration for both M/e ratios occurred. Since increasing sliding velocity increases surface temperature, the reverse trend would normally be expected. Increasing sliding velocity, however, resulted in an increase in the amount of wear particles generated. With an increase in the mass of wear particles formed, a greater quantity of heat was dissipated by wear particles, and a decrease in concentration of decomposition products was observed (fig. 9).

The decomposition products formed with 25-percent-glass-filled PTFE sliding on 440-C is shown in figure 10. With the 25-percent-glass filled PTFE composition, the ion concentration for M/e 19 ( $F^+$ ), M/e 31 ( $CF^+$ ), and M/e 50 ( $CF_2^+$ ) was found to increase with increasing sliding velocity. This result was not like that obtained with unfilled PTFE, where very little M/e 19 ( $F^+$ ) was observed. With the 25-percent-glass-filled PTFE, M/e 19 ( $F^+$ ) was the principal decomposition product detected by the mass spectrometer. This result indicated that carbon to fluorine bond cleavage must have occurred at the sliding interface. The wear with glass-filled PTFE was 100 times less than that obtained with unfilled PTFE, and the heat, therefore, was not carried away as readily from the interface. A thin film of PTFE may have been interposed between the metal surface and the glass fibers. As a result of the thin film present, it is hypothesized that localized decomposition of the PTFE film occurred and increased with an increase in sliding velocity or surface temperature. The presence of carbon on the glass-filled PTFE rider surface was observed, and only a small concentration of carbon was observed in the mass spectrometer tracing. This indicated that the decomposition was taking place at the sliding interface and not in the mass spectrometer. If decomposition had occurred in the mass spectrometer by carbon to fluorine bond cleavage, an increase in fluoride ion concentration would be accompanied by the increase in carbon ion concentration, but this was not observed. It must be pointed out, however, that some cleavage of the carbon to fluorine bond should be expected in the mass spectrometer.

In the sliding velocity experiments conducted with copper-filled PTFE, very small concentrations were observed for the two M/e ratios, 19 and 31, over the range of sliding velocities investigated (fig. 11). These results indicate that the decomposition of the PTFE was slight. This effect cannot be related to wear because the wear was the same for both glass- and copper-filled compositions (fig. 8). With copper, it is believed that a similar mechanism prevailed as was observed with glass; that is, a thin film of PTFE was interposed between the copper particles and the metal disk surface functioning as a lubricant. Unlike the glass fillers, however, copper is a good heat conductor. It serves to conduct heat away from the interface. The copper dissipates the heat generated and reduces the decomposition of PTFE. Although there is only 25-percent copper nominally present in the PTFE composition at the sliding interface, the discrete particles flow out, and the exposed area of interface copper increases (see bright areas of photographic insert of fig. 11). (Thermal conductivity at 68° F in Btu/(hr)(sq ft)(°F)(ft) for PTFE is 0.118; for copper, 226.)

Since heat rejection appeared to play an important role in the decomposition of PTFE, some experiments were conducted with a 15-percent glass composition sliding on (1) aluminum oxide (a poor heat conductor), (2) silver, and (3) copper (both good conductors) to determine the influence of the thermal conductivity of the disk material on the wear characteristics observed for the glass-filled PTFE compositions. The friction and wear results were similar for aluminum oxide and copper disk materials.

A comparison of the mass spectrometer data showed, as might be anticipated, that the concentration of the polymer degradation products was significantly less with the copper disk than with the aluminum oxide disk. From the preceding discussion, it becomes apparent that the use of materials with high thermal conductivity for mating surfaces as well as for fillers of polymers can contribute significantly to the stability of the polymers during sliding.

The data for silver were not meaningful for the experiment mentioned above because the silver transferred and adhered to the PTFE rider specimen. The friction data obtained were characteristic of silver sliding on itself. The capability of silver to adhere strongly to other completely dissimilar materials is worthy of special note.

Because of the encouraging results obtained in evaporation studies with the polyimide composition (fig. 4), some friction and wear studies were conducted in vacuum ( $10^{-9}$  mm Hg) with polyimides sliding on both metals and on itself. The results obtained in these experiments are presented in figure 12. In figure 12 the friction and wear for polyimide riders on 440-C stainless-steel disks, with the polyimide cut in two directions from the parent material block, are compared. Any orientation effects are of negligible magnitude. Although the friction for polyimides on 440-C stainless steel was relatively low compared with PTFE, the wear to the polyimide rider was somewhat higher than for the filled PTFE compositions (fig. 8). If these results are compared with those in figure 8 for unfilled PTFE, however, the wear for the polyimide is only 1/15 of that obtained with PTFE, and the friction is also lower. The unfilled polyimide also had better wear and friction properties than the filled PTFE compositions.

The friction coefficient obtained with polyimide sliding on polyimide was much higher than that obtained with polyimide sliding on 440-C stainless steel. The wear with polyimide on polyimide, however, was 1/500 of that obtained with polyimide on metal. The polyimide disk specimen did, however, show evidence of wear. With the metal disk, a thin nearly transparent polyimide film was transferred to the disk surface. The film was not thick enough to be detected in surface profile traces (magnification  $\times 1000$  in the vertical direction). It prevented, however, any measureable wear to the metal disk surface.

A 15-percent graphite-filled polyimide composition was also examined in friction and wear studies (fig. 12). The presence of graphite in the polyimide did not improve its lubricating characteristics in vacuum; relatively high friction and wear were obtained. Friction and wear experiments were also conducted with a 20-percent-copper-fiber-filled polyimide composition. In these experiments the filled polyimides slid on 440-C stainless steel. Both friction and wear for the copper-fiber-filled composition were lower than those obtained with the unfilled composition. The friction and wear obtained with 20-percent-copper-fiber-filled polyimide were lower than those obtained with 25-percent-copper-powder-filled PTFE (fig. 8) or any other compositions containing PTFE or PCFE.

The results obtained in this investigation with polyimides and PTFE in vacuum indicate the polyimide to be a superior material to PTFE in friction, wear, and outgassing characteristics at modest temperatures.

The friction and wear characteristics were determined for the two epoxy compositions examined in the evaporation studies of figure 5. The experimental conditions were the same as for the other polymers in obtaining the comparative data of figures 8 and 12. The epoxy MoS<sub>2</sub> composition sliding on 440-C stainless steel produced a friction coefficient of 0.015. The wear to the rider specimen was also very low ( $2.75 \times 10^{-7}$  cu in./hr). The epoxy MoS<sub>2</sub> composition had friction and wear as well as evaporation properties that are at least comparable with the polyimide and superior to the PTFE compositions reported herein. The epoxy PTFE composition exhibited very low friction. The specimen, however, swelled, changed color, and developed surface cracks, which indicated that thermal decomposition had occurred.

#### Friction and Wear of Binary Alloys

The tendency for clean metals or alloys to weld in vacuum was demonstrated in reference 3. The conventional bearing steel 52100 welded readily in vacuum once residual surface oxides and contaminating films were worn away. Mass welding or seizure of metal surfaces did not occur in the presence of surface oxides and other films; based on these results, an approach that can be taken to avoid such welding would be the addition of oxides to the metal structure. Thus, a continuous supply of metal oxide would be available from the structure to replace surface films that are worn away. Initial study of this concept was with binary alloys.

Electrolytic nickel was used as a base metal, and nickel oxide (NiO) was incorporated in the structure. Various percentages of the oxide were incorporated in the structure of electrolytic nickel. The nickel oxide percentages ( $\pm 0.08$  percent) of each alloy, is presented in table I.

A hardness survey of the surface of the nickel - nickel oxide alloys indicated an average Rockwell hardness of B61 in the nickel matrix. In the areas of finely dispersed nickel oxide, there was a slight hardness increase. In those areas where large nickel oxide particles segregated, the hardness approximated Rockwell B85. The individual oxide particles were too small for hardness checking.

The friction, wear, and welding characteristics of these alloys were next determined in vacuum, and the results obtained are presented in figure 13. All the experiments of figure 13 were started at an ambient pressure of  $10^{-9}$  mm Hg; however, considerable outgassing in some of the experiments occasionally raised the pressure to  $10^{-8}$  mm Hg.

The coefficient of friction obtained with electrolytic nickel was high ( $f = 2.42$ ) and erratic. During the principal portion of the experiment, considerable welding and weld breaking occurred and resulted in rapid changes in friction readings. Wear measurements to the rider speci-

men after the experiment indicated a wear rate of  $5.28 \times 10^{-3}$  cubic inch per hour. A true measure of wear under operating conditions as described in this experiment is extremely difficult to achieve, because metal is being continuously transferred back and forth from one specimen to another. Examination of both disk and rider surfaces after the experiment revealed macroscopic pieces of transferred material on both specimen surfaces.

The addition of various percentages of nickel oxide to electrolytic nickel reduced both friction and wear of nickel in vacuum (fig. 13). The coefficient of friction for the nickel - nickel oxide alloys ranged from 0.5 to 0.8. This friction is in the range of that which might be experienced with nickel-base alloys in air at atmospheric pressure. The rider wear decreased to less than half that experienced with electrolytic nickel. The transfer of large quantities of metal from one surface to another decreased with increase in nickel oxide content. From the results with these alloys, the addition of nickel oxide to nickel seemed to decrease the welding tendency in vacuum and to reduce friction and wear to a limit that would be experienced for nickel-base alloys in air at atmospheric pressure.

Although the addition of nickel oxide to electrolytic nickel improved performance in vacuum, friction and wear were still high. Consideration was therefore given to the production of a structure that would be self-lubricating. Examination of the physical properties of various soft metals indicated that tin might be a desirable metal for vacuum lubrication applications. Tin has a low evaporation rate in vacuum, has good lubricating characteristics as a thin film, and, when alloyed with a base metal like nickel, will produce a duplex structure. This duplex structure consists of a hard matrix with a dispersed soft phase. Various percentages of pure tin were therefore alloyed with electrolytic nickel. A nickel-tin compound ( $\text{Ni}_3\text{Sn}$ ) appeared in the grain-boundary region with the addition of 5 percent tin to nickel; the compound was identified by X-ray diffraction. As the percentage of tin in the alloy was increased, a continuous increase in concentration of the nickel-tin compound occurred along the grain boundaries. Hardness testing of the alloy surface revealed a general increase in alloy hardness with increasing tin content as shown in the following table:

Tin in nickel-tin alloy composition, percent	5	10	15	20	25
Rockwell hardness	B67	B77	B80	B85	B106 (C30)

In all structures, the nickel-tin compound had an average Rockwell hardness of C50. The relative hardness of nickel matrix and addition phases will be discussed later.

The friction and wear characteristics of the nickel-tin alloys were next determined in vacuum ( $10^{-9}$  mm Hg), and the results are presented in

figure 14. The addition of 5 percent tin to nickel was sufficient to decrease the coefficient of friction from 2.42 to 0.22 (a factor of approx. 11). The wear decreased to one-fifth that obtained with pure nickel. These results demonstrate that a duplex structure, when one phase is capable of surface flow, is extremely effective in reducing friction and wear. The coefficient of friction was approximately the same for 5, 10, 15, and 20 percent tin. For 25 percent tin, an increase in friction was observed that indicated that there may be an optimum in phase concentration necessary to achieve the minimum in friction coefficient. There was, however, a continuous decrease in wear with increased tin content. (The wear decreased from  $5.28 \times 10^{-13}$  cu in. for electrolytic nickel to  $0.01 \times 10^{-3}$  cu in. for 25-percent-tin-nickel alloy or a decrease in wear by a factor of more than 500.)

Sulfur has been used for some time as an additive to steels to improve ease of machining these alloys (hence, the term "free machining"). It has also been used in various compounds as an extreme pressure additive to oils and in reactive gas lubricants (e.g., sulfur hexafluoride). It was therefore decided to incorporate sulfur in the structure of pure electrolytic iron, to determine the influence of sulfur on the friction and wear characteristics of iron in vacuum. Since sulfur is soluble within the iron structure in very small amounts only, any sizable additions of sulfur to iron should result in segregation of the sulfur, as iron sulfide, along the grain boundaries.

Various amounts of sulfur were added, as iron sulfide, to electrolytic iron. The sulfur was present as iron sulfide within the iron structure as well as along the grain boundaries for all sulfur-containing melts. The considerable amount of sulfur in the 0.45-percent iron-sulfur alloy resulted in a structure that has been described as a "cell membrane" or enveloping structure because of the pattern shown by iron sulfide in the vicinity of the grain boundaries.

Some vacuum friction and wear experiments were next conducted with these iron-sulfur alloys, as well as electrolytic iron, and the results obtained are presented in figure 15. With electrolytic iron (no sulfur), considerable welding and mass transfer occurred; an accurate measure of friction and wear was difficult. The addition of less than 0.1 percent sulfur to iron resulted in a decrease in the welding tendency for the alloy at  $10^{-9}$  mm Hg. Further additions of sulfur to iron gave a coefficient of friction less than 0.3. The welding tendency of the iron-sulfur alloys was observed visually to decrease with increasing sulfur content; this result is illustrated by figure 16.

The addition of sulfur to metals, in general, tends to accelerate oxidation. In order to determine the influence of various ambient pressures (oxygen concentrations) on the friction characteristics of the 0.45-percent iron-sulfur alloy, an experiment was conducted at ambient pressures from  $10^{-9}$  to 760 mm Hg. With sulfur present as an oxidation catalyst as well as separate film former, there was a sufficiently thick

film present at all pressures that friction did not change markedly over the pressure range from  $10^{-9}$  to 760 mm Hg. At 760 mm Hg, where the oxide film was thicker, the coefficient of friction remained less than 0.3.

Good mechanical properties and improved friction and wear characteristics are believed obtainable with other simple binary nickel alloys. Examination of the phase diagrams for the nickel alloys indicated that a binary nickel-aluminum system would be of interest. It is possible to obtain two intermetallic compounds of nickel and aluminum,  $\text{Ni}_3\text{Al}$  and  $\text{NiAl}$ . Based on the phase diagram (ref. 32), three alloy compositions were prepared with the intermetallic  $\text{Ni}_3\text{Al}$  in a nickel matrix. A fourth composition of  $\text{NiAl}$  was obtained commercially. The compositions contained 5.5, 13.3, 16.4, and 27.1 percent aluminum and had the microstructure shown in the photomicrographs of figure 17. These compositions are three alloys with an increasing concentration of intermetallics in solid solutions (duplex structure) and one with a simple intermetallic  $\text{NiAl}$  (27.1 percent aluminum).

Friction and wear experiments were conducted in vacuum with the four nickel-base alloys. The results obtained in these experiments are presented in figure 17. The friction coefficient did not decrease with the increase in the  $\text{Ni}_3\text{Al}$  compound obtained in going from 5.5 to 13.3 percent aluminum. At 16.5 percent aluminum, however, a decrease in friction was observed. The rider wear continued to decrease with an increase in percentage of intermetallic present. When, however, sufficient aluminum was added to the alloy so that only the intermetallic compound  $\text{NiAl}$  was present, a further slight reduction in friction was observed, but the rider wear and transfer increased. Transfer to the disk surface decreased with increasing aluminum content for the two-phased structure. Essentially no metal transfer was obtained in vacuum with the simple binary alloy and the best composition was 16.4 percent aluminum in nickel, which had a duplex intermetallic structure.

## Friction and Wear of Complex Ferrous

### Alloys Containing Sulfur

Because improved friction, wear, and nonwelding tendencies were obtained with binary iron-sulfur alloys, some bearing alloy compositions were cast with the addition of sulfur to their structure (table II). Photomicrographs for two of these alloys, M-2 steel and 440-C stainless steel, are presented in figure 18 together with photomicrographs for the standard alloy compositions without sulfide inclusions. The standard wrought alloys have a fine grain structure. In contrast, casting of the sulfide inclusion composition results in a very coarse grain structure as shown in figures 18(c) and (d). With the modified M-2 composition, the inclusions appear primarily in the grain boundary regions of the alloy, while with 440-C stainless steel the sulfides appear to be dispersed throughout the structure as well as at the grain boundaries. The sulfur-modified alloys were given (after machining into specimens and just before being finish ground) the same heat treatment as prescribed

for the standard alloys. The wrought alloys and the sulfur-modified cast alloy had similar hardnesses (table II).

The friction and wear characteristics of these alloys were next determined in vacuum ( $10^{-9}$  mm Hg) and the results obtained are presented in figure 19. The friction and wear experiments were conducted with the wrought standard alloys, the standard alloys recast, and the sulfur-modified cast compositions. In general, all three compositions showed an improvement in friction and wear with the addition of sulfur to the structure. The unmodified recast alloys had friction and wear properties similar to the wrought form of the same alloys. The most marked improvement in friction was noted with 52100. Initial friction coefficient values of 0.68 for the wrought structure were obtained with residual oxides present. With wearing away of surface oxides complete welding of the metal was observed. A friction value of 0.54 was obtained with the sulfur-modified structure. The greatest reduction in wear, however, was observed with 440-C stainless steel, for which a difference in wear of about 1000 times resulted from the addition of sulfur. It is of interest to note that metal transfer was observed with the unmodified M-2 and 440-C alloys. With the addition of sulfur, however, no metal transfer was noted for either of the two alloys.

The mechanism responsible for the reduction in friction, wear, and metal transfer is the formation of a protective surface film on the alloy composition by the smearing out of the sulfide phases over it. The sulfide surface film then substitutes for normal metal oxides in preventing gross metal contact from occurring.

Since the role of the sulfide in the alloy is to provide a protective surface film, some friction and wear experiments were conducted with only one of the two specimens modified by the addition of sulfur in order to determine whether it was necessary for both to contain sulfur. It was anticipated that an effective film might be provided by one specimen. With the sulfur-modified 440-C rider sliding on the standard 440-C disk material, sufficient sulfide was continuously present at the metal interface to reduce the friction from 0.66 to 0.40; the amount of sulfide present, however, was not adequate to reduce the rider wear appreciably. When the specimen combination was reversed (standard 440-C rider on sulfur-modified disk), the friction was also about 0.4; the rider wear, however, decreased to a value near that which was obtained with a sulfur-modified rider on a sulfur-modified disk.

Experiments were conducted in vacuum with standard and sulfur-modified M-2 to determine the effect of the sulfide film on friction wear, and surface failure over a range of sliding velocities to nearly 2000 feet per minute. There was no significant change in the relative effectiveness of the sulfide film at the various sliding velocities.

Higher friction was obtained with sulfur-containing complex alloys than with sulfur-containing iron. This result can probably be explained by the presence of sulfides other than FeS in the complex alloys.



## Friction and Endurance of MoS<sub>2</sub> Solid Film

### Lubricant Coatings

The inorganic compound MoS<sub>2</sub> is known to have good lubrication properties in vacuum and is widely used (ref. 1). A very common method of using MoS<sub>2</sub> is in bonded solid films. Such films have a finite endurance life that is very much a function of the bonding material. Many friction and wear experiments have been run at a pressure of  $10^{-9}$  mm Hg with a load of 1000 grams on a 3/16-inch-radius rider specimen contacting the coated flat surface of a rotating disk specimen (fig. 2). Low friction ( $f = 0.02$  to  $0.08$ ) characteristic of MoS<sub>2</sub> alone is usually obtained but the bonding material (e.g., ceramics) in some instances does have an adverse effect on friction.

The endurance characteristics of bonded MoS<sub>2</sub> films are variable and depend significantly on both the bonding media used and the application technique. More consistent data have been obtained with commercial coatings applied in the laboratory than when applied by the commercial sources. Coatings employing silicones, ceramics, or metal matrix bonding agents have shown lower endurance lives than phenolic-epoxy or sodium silicate formulations. The former materials would not usually survive 1 hour of operation (42,000 cycles) in the friction and wear experiment. The phenolic-epoxy and sodium silicate bonded films have been the most durable of the coatings with endurance lives approaching 1 million cycles in these experiments. Both of the latter coatings have been used successfully on the S-Monel retainers of ball bearings run in vacuum ( $10^{-9}$  mm Hg) for periods of about 50 hours. These size-204 angular-contact bearings (the support bearings of fig. 2) were operated at 800 to 4000 rpm with 50 pounds thrust load on one of them. Recently, bearings employing retainers made of molded polytetrafluoroethylene with glass fibers plus MoS<sub>2</sub> as fillers have also been used successfully in this application.

Coatings applied using bonding agents adhere largely because of mechanical bonding. Severe thermal shock such as quenching in liquid nitrogen causes the coating to be more subject to mechanical failure. Matching of thermal-expansion characteristics and improved heat transfer of films by using metal fillers in the bonding media has been studied by several organizations. Recent experience with metal fillers in bonded solid lubricant film was reported favorably for use in vacuum (ref. 25).

Coatings have also been explored that utilize a chemical bond for adherence. One coating is obtained by forming a metallic molybdenum film on a metal surface and subsequently reacting that surface with a sulfur-containing media in a retort to achieve chemically bonded molybdenum sulfide coating.

Another complex coating formed by vacuum deposition and chemical reaction utilizing an electron beam source for activation energy has also been considered. Limited experience with such coatings in the friction apparatus previously described did not show them to be superior to the previously described coatings.

A common method of applying  $\text{MoS}_2$  to metal surfaces is by varied burnishing techniques. One burnishing method has been utilized at the Lewis Research Center that has some distinguishing features. In that method, rigorously cleaned (organic free) metal surfaces and a clean soft steel wire brush were charged with very fine particles of  $\text{MoS}_2$  in a previously partially evacuated and purged dry box filled with argon. The wire brush was a rotating type and was driven with a small electric drill. Burnishing by holding the rotating wire brush firmly against the metal surface in the presence of  $\text{MoS}_2$  and argon achieved at the interface a chemical bond. Sufficient energy for activation was provided by friction of the rotating wire brush with the coated surface. The argon atmosphere prevented oxidation of the  $\text{MoS}_2$  so that a more desirable reaction could occur. Electron diffraction examination of steel surfaces burnished in this manner showed the surface film to be a complex mixture of iron and molybdenum sulfides. Interference coloration was used as a control measure to obtain a burnished film that was about 500 Å thick. Pretreatment of the surfaces by vapor and glass bead "blasting" was found advantageous. Glass beads presumably did not remove surface metal but did change the character of the surface. This observation suggests that mechanical bonding as well as chemical bonding is helpful.

The friction coefficient for this burnished film ( $f = 0.3$ ) was found to be higher than for most  $\text{MoS}_2$  films ( $f = 0.02$  to  $0.08$ ). That result was a function of the greater shear resistance of the iron sulfide containing film than the usual  $\text{MoS}_2$  film. The endurance characteristics of the film were found, however, to be exceptionally good and never less than the best of the more complex bonded films. The favorable endurance characteristics have been further verified in some small-amplitude oscillating-ball-bearing experiments in vacuum. The film formed is so thin ( $2 \times 10^{-6}$  in.) that it can be applied to all the component parts of most rolling-element bearings without causing a tolerance problem.

Experience at Lewis Research Center with  $\text{MoS}_2$  coatings has long showed that the presence of an organic lubricant or other liquids had an adverse effect on endurance life. It has been considered that the fluids preferentially wet the base metal and any free particles of solids and allow the free particles to be displaced from the load bearing area. It was considered probable that a solid film having a chemical bonding with the base metal would be less subject to the adverse effect of the liquid on endurance life. Runs were therefore made with the burnished  $\text{MoS}_2$  described previously but adding a small amount of a mineral oil known to have low vapor pressure. The burnished  $\text{MoS}_2$  film was wetted with 10 drops of a 5 percent solution of mineral oil in xylene. After the xylene visibly seemed to have evaporated, the disk specimen was assembled in the friction apparatus and held under vacuum for 24 hours (at  $10^{-8}$  mm Hg) prior to the test, which was conducted at  $10^{-9}$  mm Hg. As mentioned previously, the dry burnished film showed good endurance life when precoat-ing treatment was by either vapor blast or glass-bead blast methods. Figure 20 shows typical burnished specimens after experimental runs in vacuum. When no organics were added to the burnished disk surfaces, 1-hour test periods were completed with no evidence of failure. With the

disk specimens that had been wetted with the organic liquids, however, the lubricating film failed almost immediately (in less than 2 min). A darker appearance of the specimens (see fig. 20) made it quite apparent that some residual organic material was present after the vacuum experiments at  $10^{-9}$  mm Hg. These results suggest that organic liquid should not be used with MoS<sub>2</sub> solid film coatings even when a chemical bond is achieved.

#### SUMMARY OF RESULTS

From the data obtained in this investigation with various lubricating materials in vacuum evaporation, friction, and wear studies, the following summary remarks can be made:

1. The quantity and nature of decomposition products formed with polymers sliding on various materials is determined both by the mating material and the polymer composition. Variation of the filler material in polymers can result in differences in the decomposition mechanism of the polymer. With glass-filled polytetrafluoroethylene (PTFE) compositions, sufficient heat is generated at the sliding interface to result in carbon to fluorine bond cleavage. The presence of copper as a filler in PTFE (a good heat conductor) reduces the decomposition observed.

2. The friction and wear for a copper filled polyimide and an epoxy MoS<sub>2</sub> composition indicate that as slider materials they are superior to PTFE in vacuum at ambient temperatures. Both the polyimide and the epoxy MoS<sub>2</sub> composition had lower evaporation than PTFE in their useful temperature ranges.

3. The addition of 0.4 to 0.5 percent sulfur to electrolytic iron, 52100, 440-C, and M-2 tool steel appreciably reduced surface welding normally encountered with these alloys in vacuum ( $10^{-9}$  mm Hg) although friction was not markedly reduced. Furthermore, it did reduce wear more markedly for 440-C than for the other alloys.

4. Simple binary nickel-aluminum alloys were prepared, and one was found to have superior friction and wear properties. Of the nickel-aluminum alloys examined, a 16.4 percent aluminum in nickel composition (duplex structure) exhibited the most promising behavior for vacuum applications.

5. The presence of tin in electrolytic nickel reduced both friction and wear. The friction decreased by a factor greater than 10, and the wear decreased by a factor greater than 500.

6. The presence of oxygen as nickel oxide in electrolytic nickel improved friction and wear properties.

7. A burnished MoS<sub>2</sub> film applied in an inert atmosphere had good endurance properties but gave higher friction than the best commercially available bonded films containing MoS<sub>2</sub>. The burnishing method used achieves a chemical bond with type 440-C steel; as with other MoS<sub>2</sub> bonded films, however, the presence of an organic liquid on the film had an adverse effect on endurance life.

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TABLE I. - COMPOSITION OF BINARY ALLOY CASTINGS FORMED  
IN ZIRCONIUM OXIDE CRUCIBLE

System	Alloying agent	Amount of agent added, percent	Concentration of agent in finished specimens, percent
Nickel - nickel oxide	Nickel oxide	1.50	1.35
		2.00	1.77
		2.50	1.99
		3.00	2.52
		5.00	4.73
		6.50	6.08
Nickel-tin	Tin	5.40	5.03
		10.80	9.65
		16.25	15.35
		21.0	19.65
		26.50	25.21
Iron - iron sulfide	Sulfur added as iron sulfide	0.020	0.011
		.040	.028
		.100	.054
		.250	.145
		.750	.450
Nickel - aluminum	Aluminum	6.0	5.53
		14.0	13.24
		16.6	16.40
		28.0	27.10

TABLE II. - SULFUR CONCENTRATIONS IN  
MODIFIED BEARING ALLOYS

Alloy, percent	Sulfur added, percent	Sulfur in alloy, percent	Hardness, Rockwell C
440-C	0	0.02	56
S-modified 440-C	.5	.44	57.5
52100	0	.025	60
S-modified 52100	.5	.41	60
M-2	0	-----	65
S-modified M-2	.5	.5	65



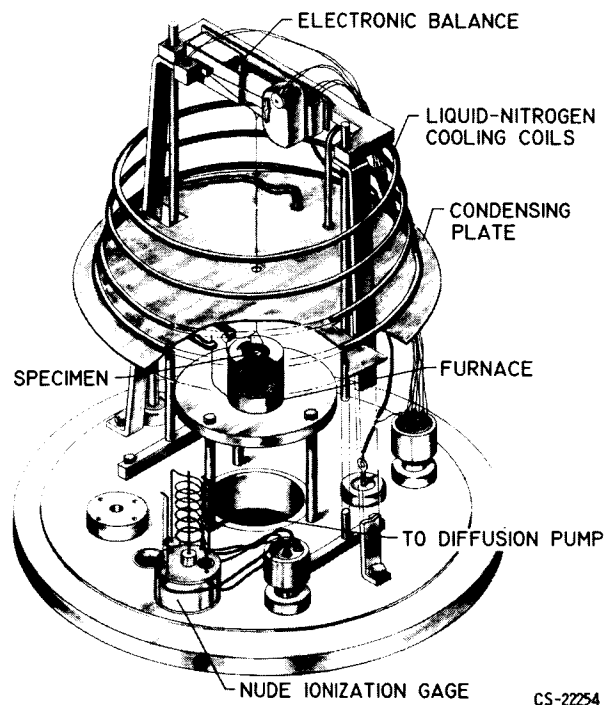


Fig. 1. - Vacuum evaporation apparatus.

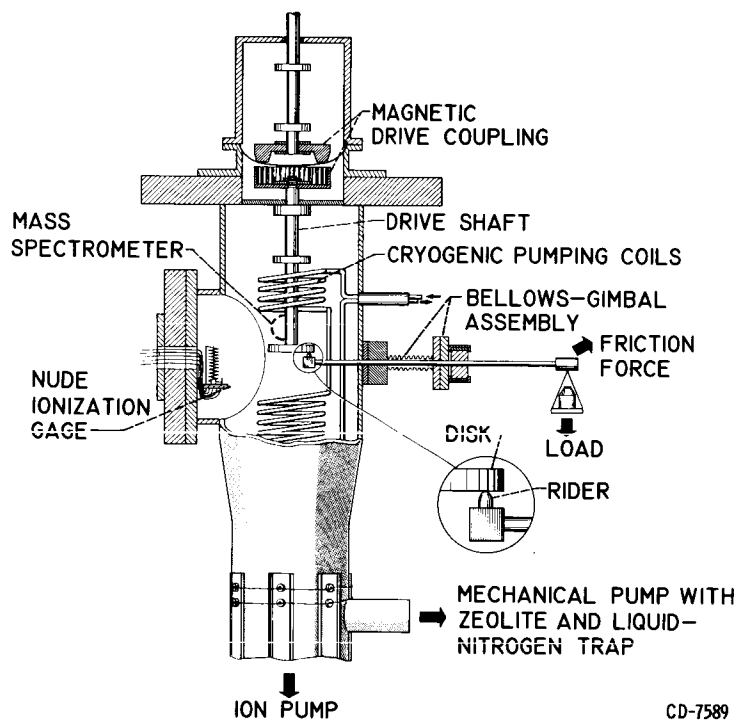


Fig. 2. - High-vacuum friction and wear apparatus.

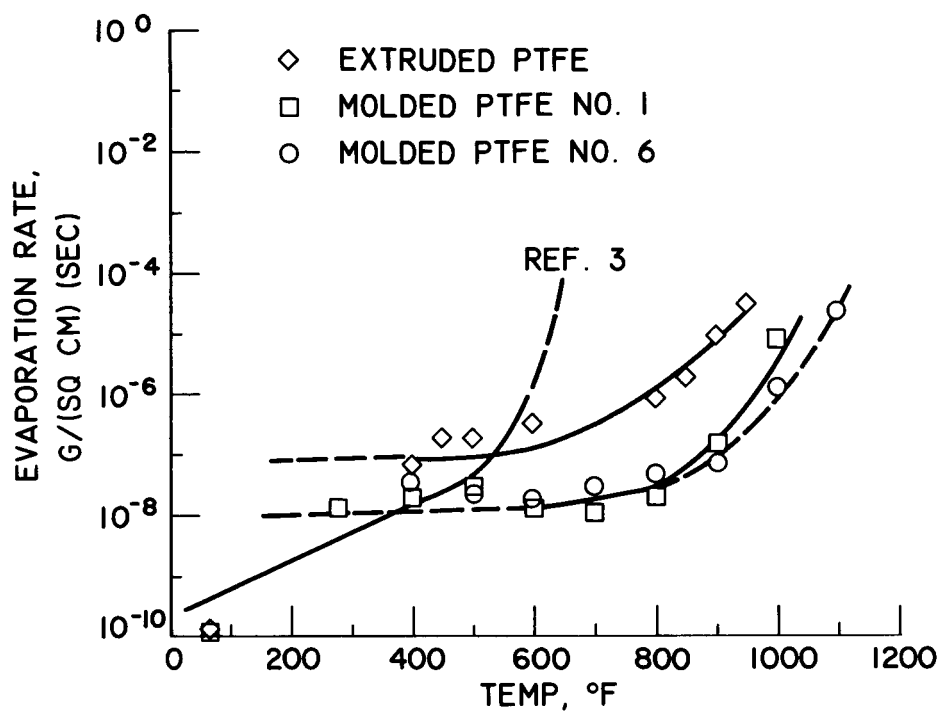


Fig. 3. - Evaporation rate for various teflon compositions in vacuum. Ambient pressures,  $10^{-7}$  to  $10^{-8}$  mm Hg.

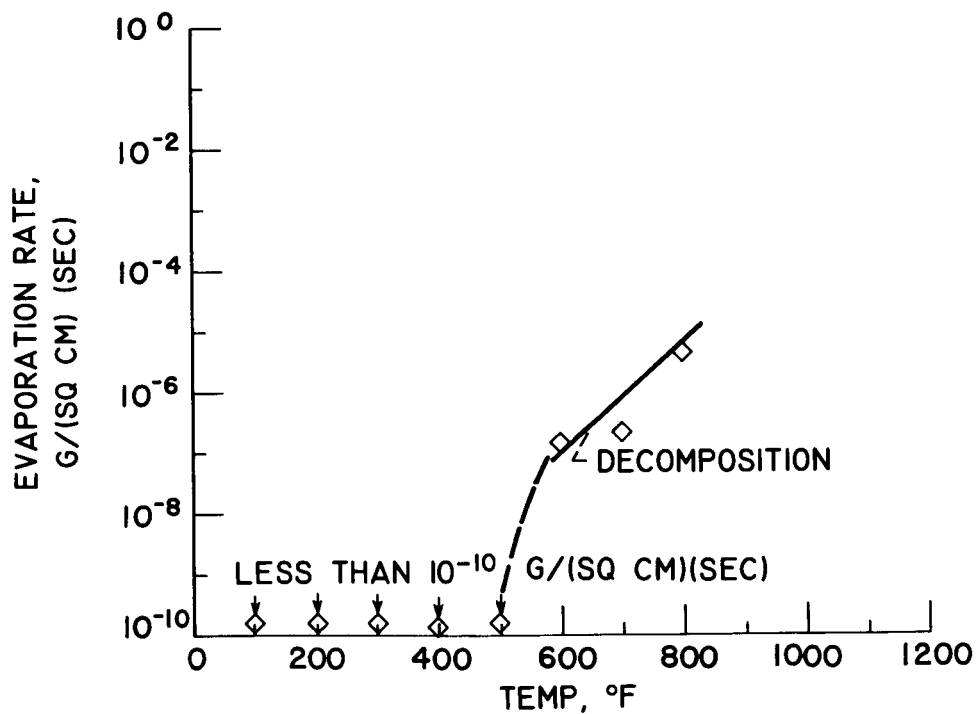


Fig. 4. - Evaporation rate of polyimide in vacuum. Ambient pressures,  $10^{-7}$  to  $10^{-8}$  mm Hg.

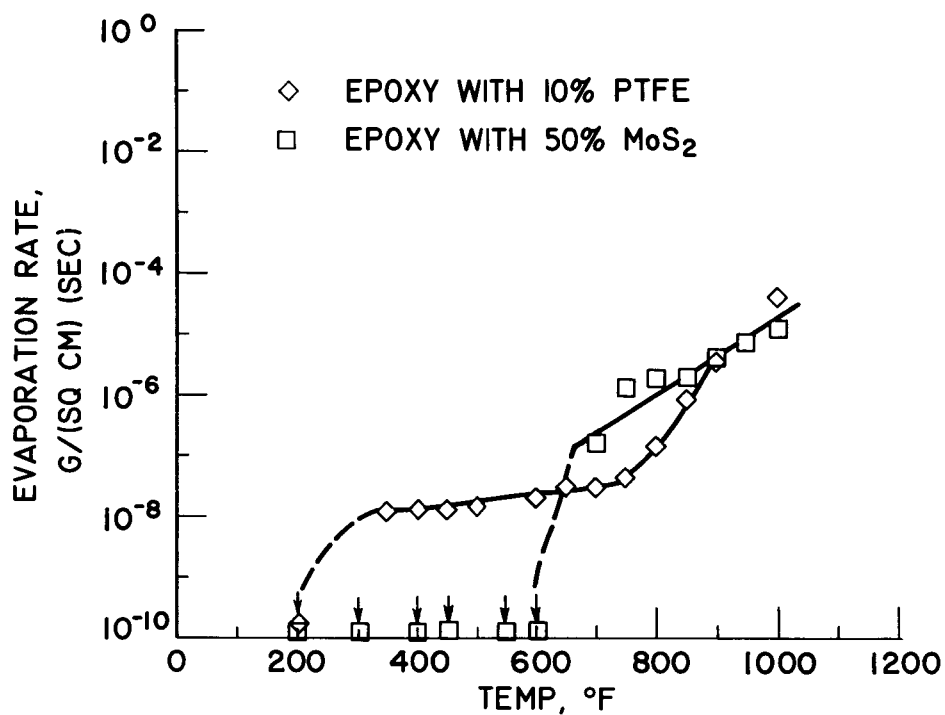


Fig. 5. - Evaporation rates for two epoxy compositions in vacuum. Ambient pressure,  $10^{-7}$  mm Hg.

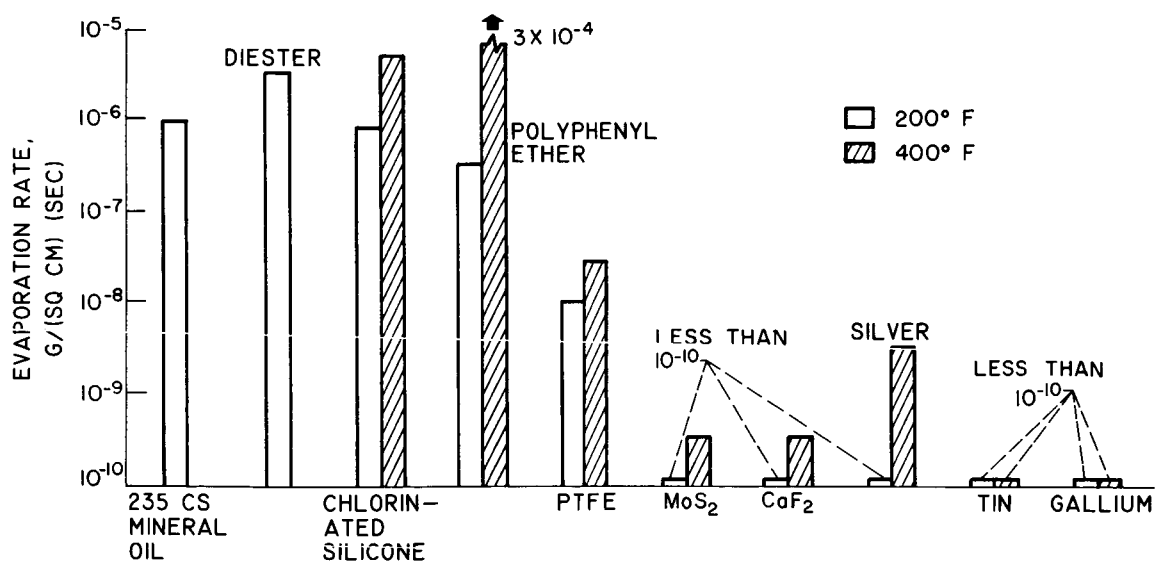


Fig. 6. - Evaporation rates for various materials in vacuum. Ambient pressure,  $10^{-6}$  to  $10^{-7}$  mm Hg.

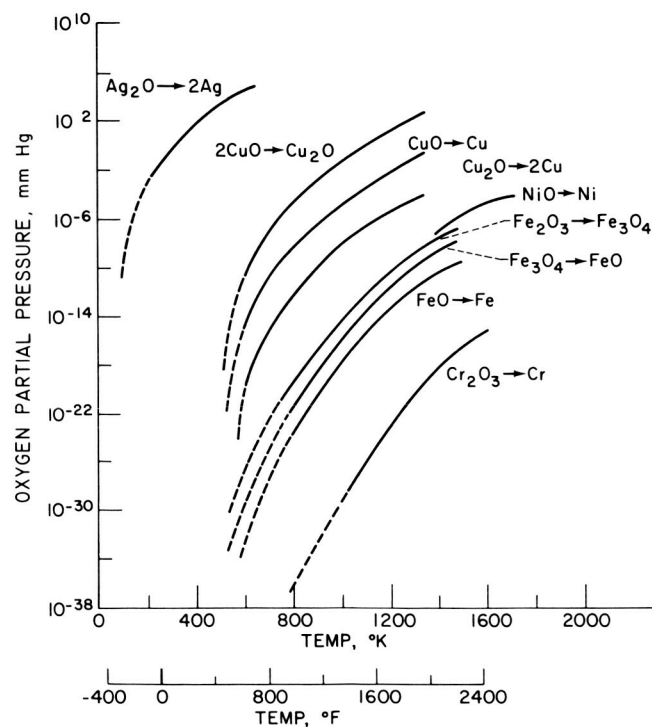


Fig. 7. - Oxygen partial pressure necessary to prevent the dissociation of metal oxides at various temperatures.

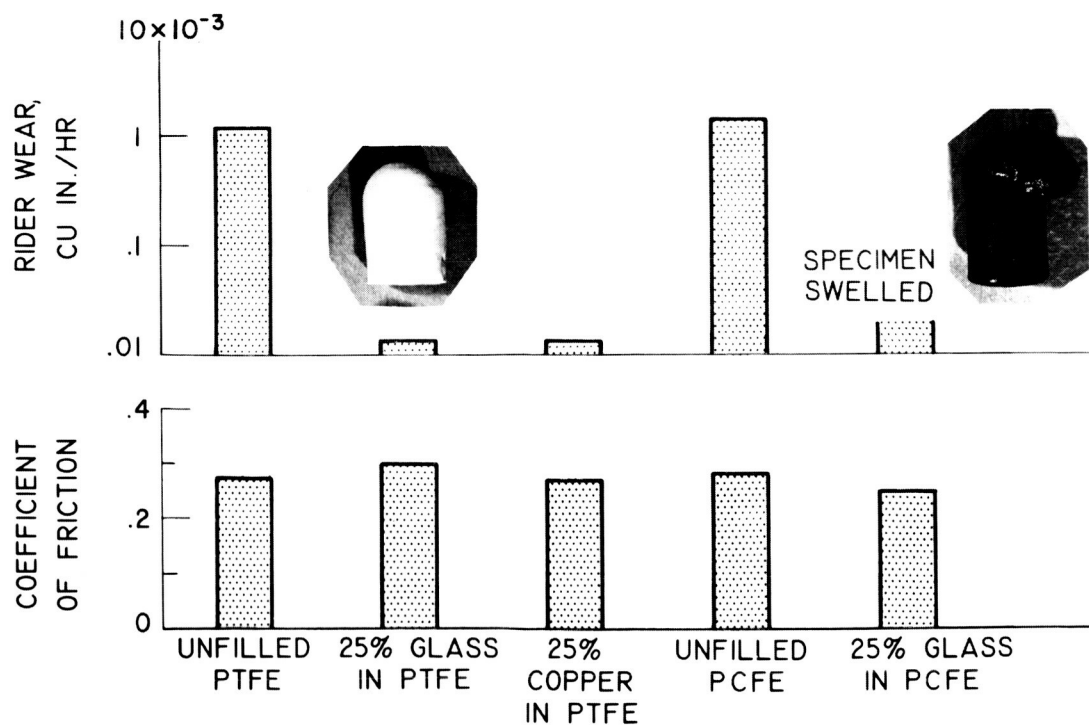


Fig. 8. - Friction and wear of PTFE and PCFE in vacuum. Disk, 440-C stainless steel; sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; no external specimen heating.

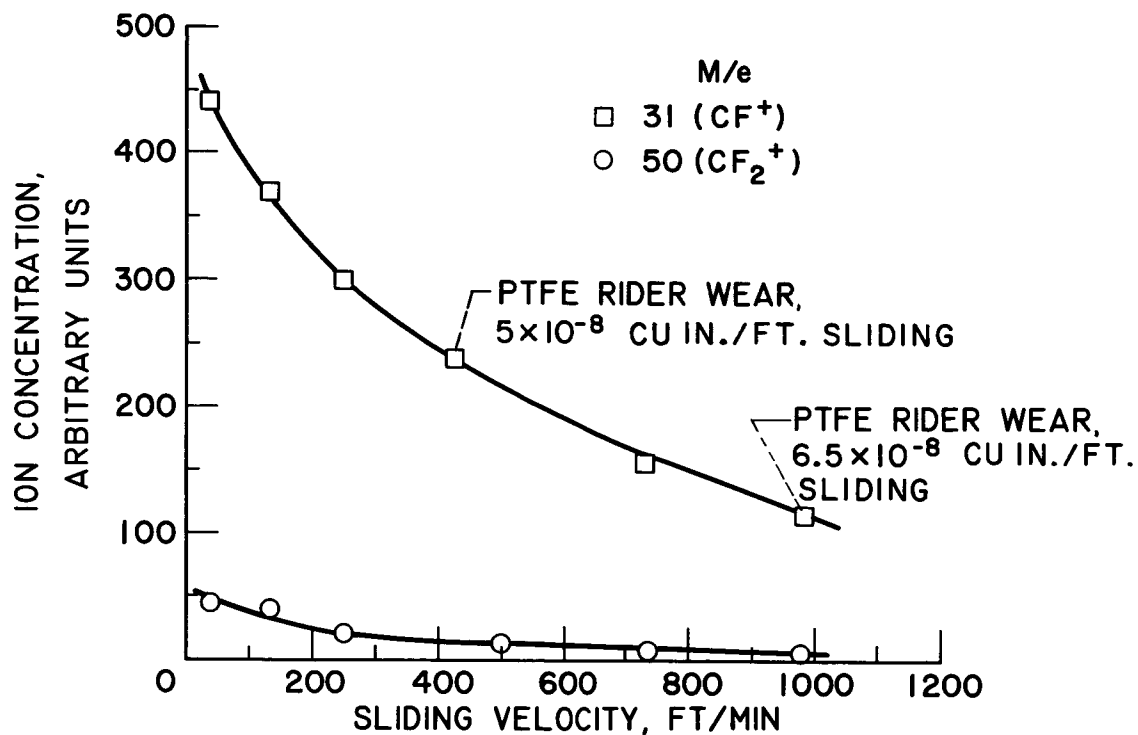


Fig. 9. - Decomposition products of unfilled PTFE obtained in vacuum friction studies. Disk, 440-C stainless steel; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; no external specimen heating.

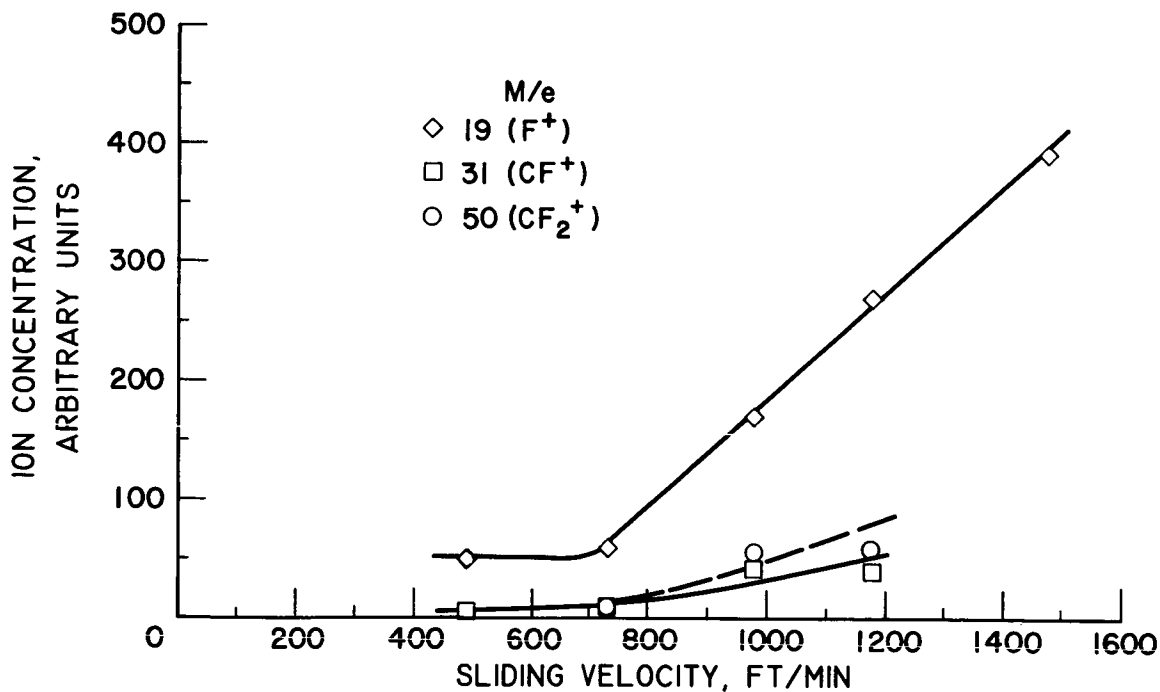


Fig. 10. - Decomposition products of 25% glass-filled PTFE obtained in vacuum friction studies. Disk, 440-C stainless steel; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; no external specimen heating.

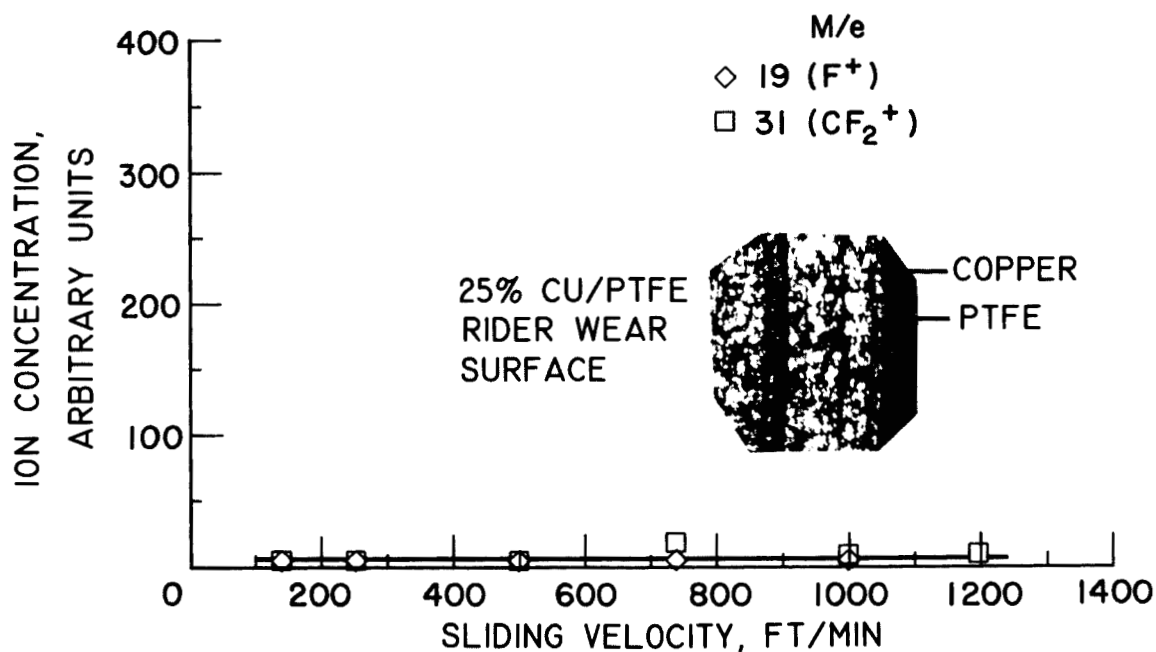


Fig. 11. - Decomposition products of 25% copper-filled PTFE obtained in vacuum friction studies. Disk, 440-C stainless steel; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; no external specimen heating.

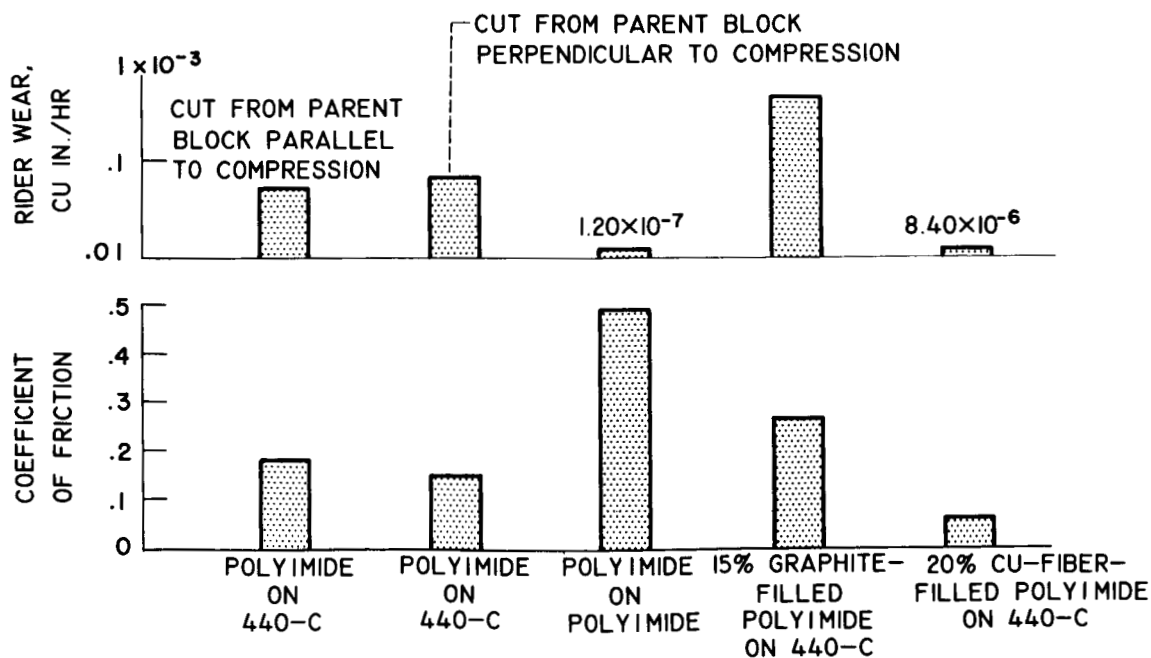


Fig. 12. - Friction and wear of polyimide on polyimide and 440-C stainless steel in vacuum. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; no external specimen heating.

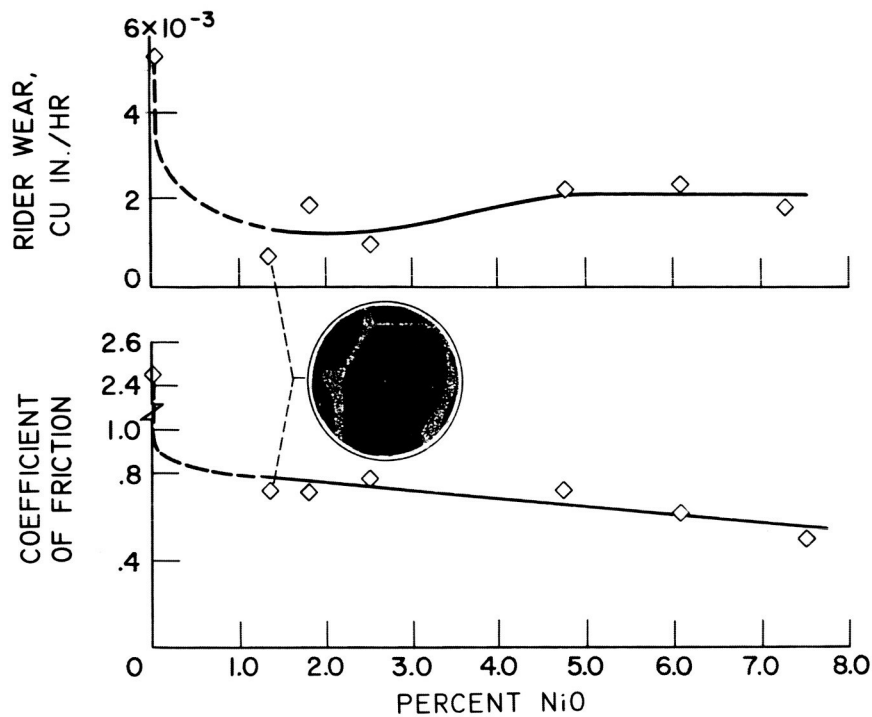


Fig. 13. - Friction and wear of nickel - nickel-oxide alloys in vacuum. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; duration of run, 1 hour.

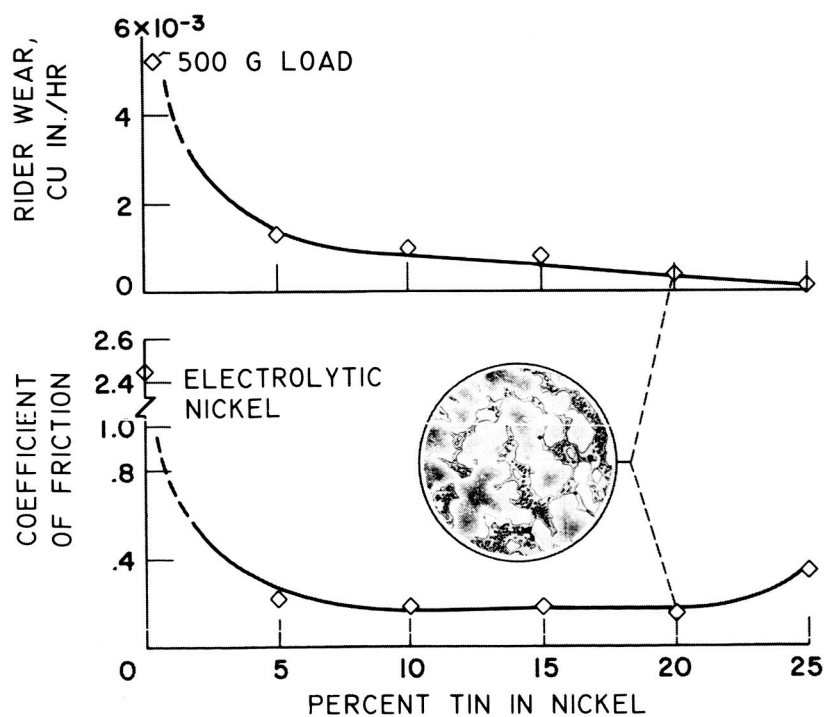


Fig. 14. - Friction and wear of tin-nickel alloys in vacuum. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; duration of run, 1 hour.

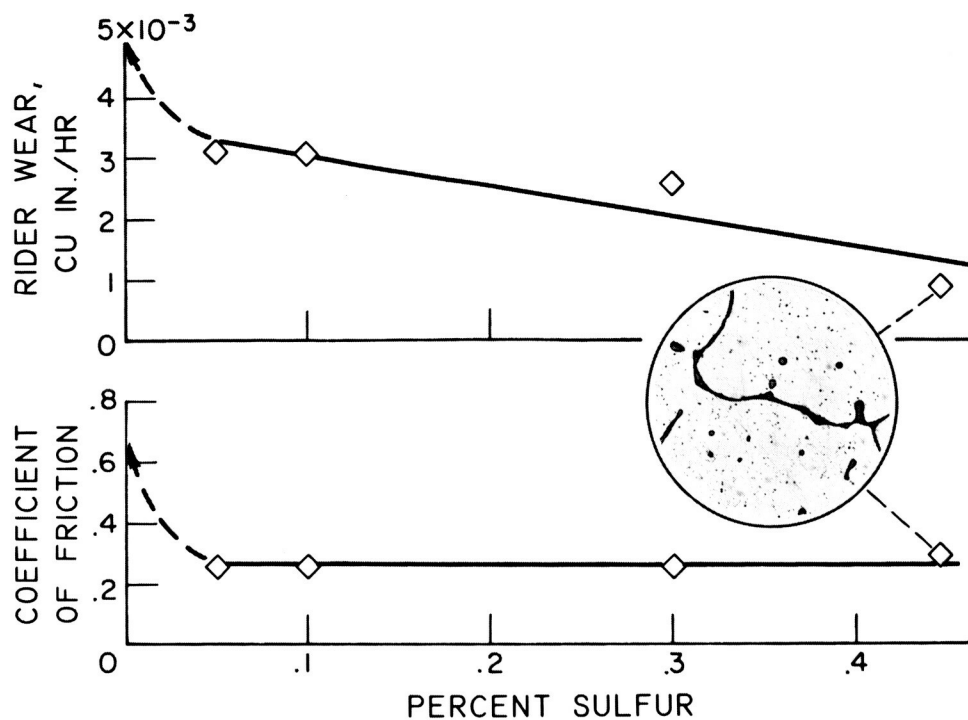


Fig. 15. - Friction and wear of sulfur-iron alloys in vacuum. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; duration of run, 1 hour.

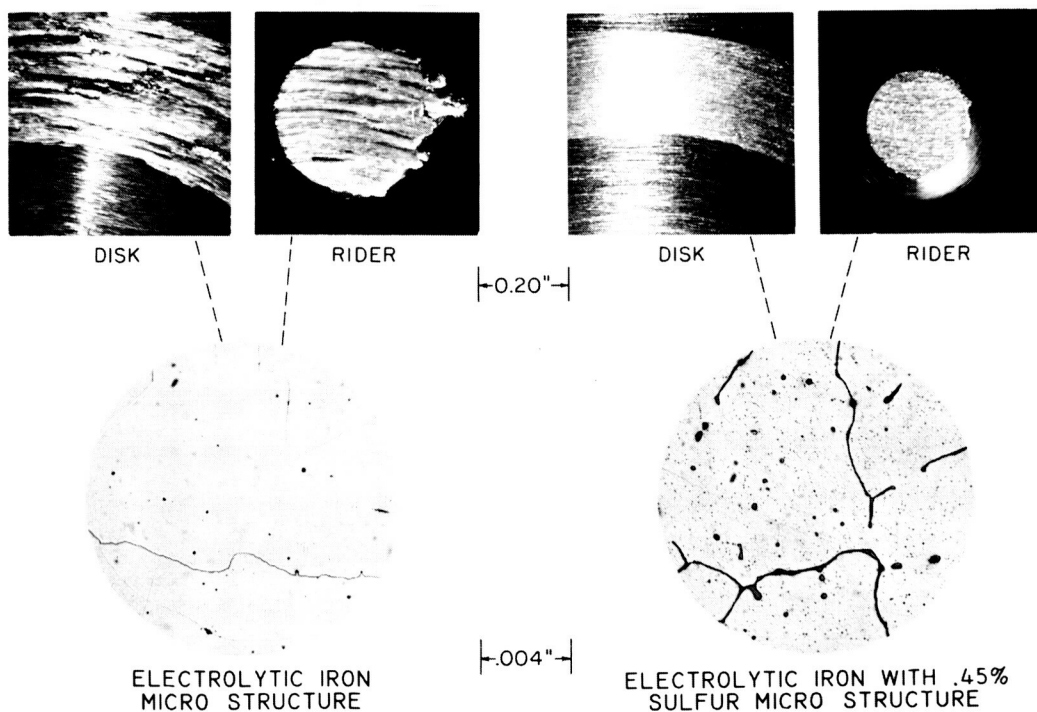


Fig. 16. - Influence of sulfur addition to iron on sliding behavior in vacuum. Ambient pressure,  $10^{-9}$  mm Hg.



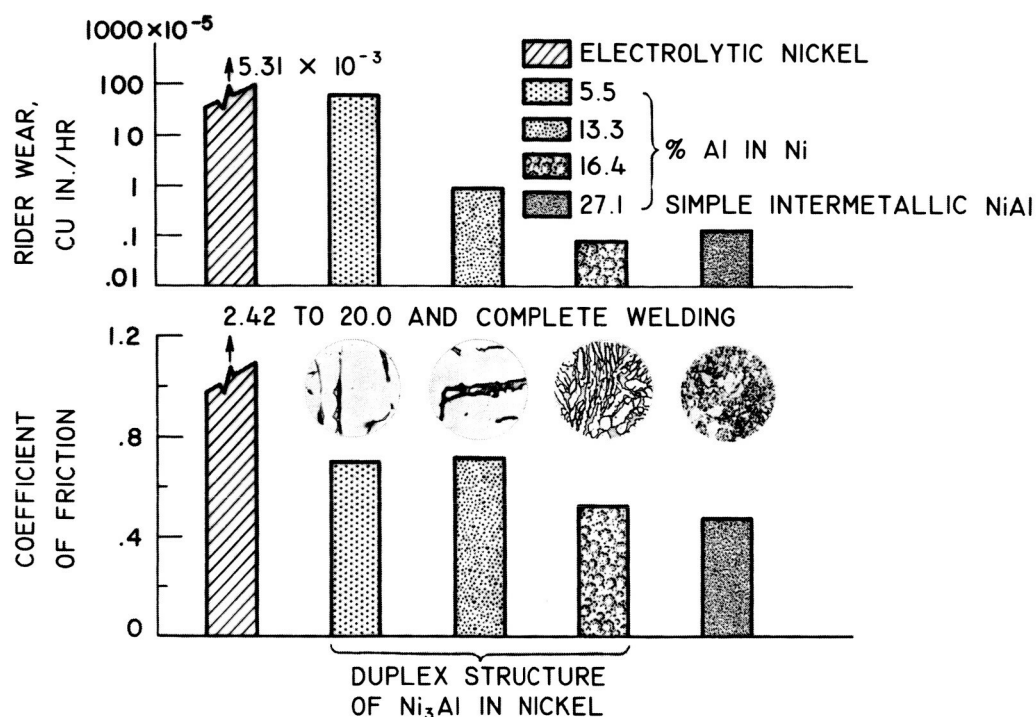


Fig. 17. - Friction and wear for nickel-aluminum alloys in vacuum. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; duration of run, 1 hour.

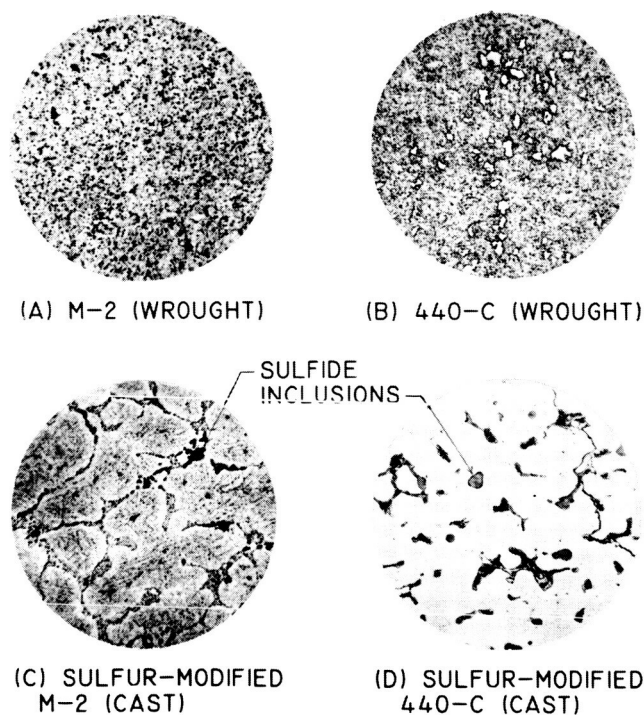
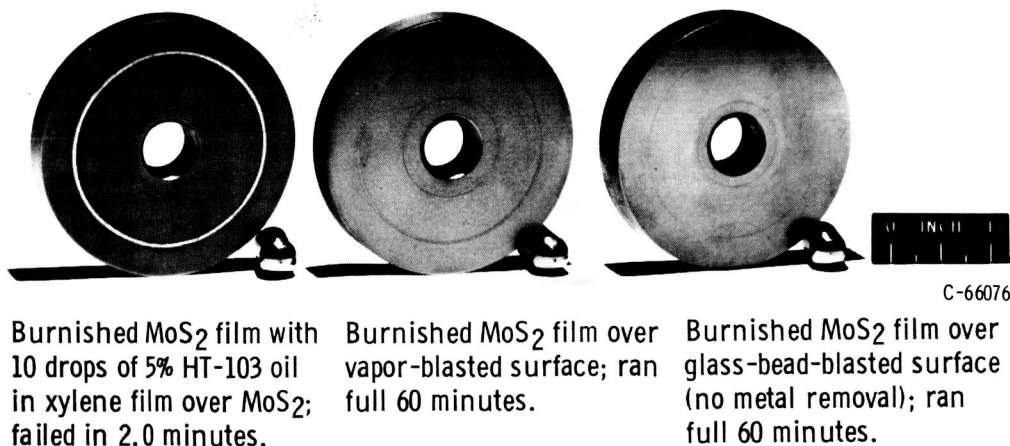


Fig. 18. - Photomicrographs of M-2 tool steel and 440-C stainless steel modified by the addition of 0.4 to 0.5 percent sulfur to the structure. 500X.



Fig. 19. - Coefficient of friction and rider wear for sulfur-modified alloys. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; temperature,  $75^{\circ}$  F; duration of run, 1 hour.



Burnished  $\text{MoS}_2$  film with 10 drops of 5% HT-103 oil in xylene film over  $\text{MoS}_2$ ; failed in 2.0 minutes.

Burnished  $\text{MoS}_2$  film over vapor-blasted surface; ran full 60 minutes.

Burnished  $\text{MoS}_2$  film over glass-bead-blasted surface (no metal removal); ran full 60 minutes.

Fig. 20. - Disk and rider specimens after operation in vacuum. Hardened type 440-C stainless steel specimens; sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure,  $10^{-9}$  mm Hg; ambient temperature,  $75^{\circ}$  F.