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SPUTTERING TECHNOLOGY IN SOLID FILM LUBRICATION

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Abstract

Potential and present sputtering technology is discussed as it applies to the deposition of solid film lubricants particularly MoS_2, WS_2, and PTFE. Since the sputtered films are very thin (2000 Å to 6000 Å) the selection of the sputtering parameters and substrate condition is very critical as reflected by the lubricating properties. It was shown with sputtered MoS_2 films that the lubricating characteristics are directly affected by the selected sputtering parameters (power density, pressure, sputter etching, dc-biasing, etc.) and the substrate temperature, chemistry, topography and the environmental conditions during the friction tests. Electron microscopy and other surface sensitive analytical techniques illustrate the resultant changes in sputtered MoS_2 film morphology and chemistry which directly influence the film adherence and frictional properties. These thin, sputtered lubricant films perform reliably only when the proper sputtered parameters and substrate conditions are selected.

INTRODUCTION

A decade has passed since MoS_2 and other solid lubricants (WS_2, $NbSe_2$, CaF_2, BN, Au, Ag, PTFE, etc.) were first sputtered as thin films on sliding or rotating surfaces. Sputtering is not a new process. The sputtering technology in depositing solid lubricants, particularly MoS_2, is however relatively new and yet, has moved very quickly from the research laboratory into practical industrial use. Sputtered MoS_2 lubricant films are most widely used in the aerospace and aircraft industries (1-3). To cite a few applications: high speed, long-endurance bearings in gyroscopes and accelerometers, gears and bearings for spacecraft harmonic drive assemblies, and despin bearings in communication spacecraft. Presently tests are being conducted for sputter coated bearings for Minuteman missile and intercontinental missile programs. Many industries are also rapidly turning to sputtered lubricant films where they show distinct improvement and advantages over other lubricating methods. These include, for example, sputtered MoS_2 bearings for rotating anode X-ray tubes which operate in high vacuum and at elevated temperatures, sputtered solid lubricants for videotapes and also for long playing records.

Generally, sputtering is of invaluable importance where high precision components having close tolerances are used, wear debris formation is critical, extremely thin, uniform films without a binder have to be used and high reliability requirements have to be satisfied. Sputtering is primarily used as a thin film deposition technique (films <1 μm). In tribological applications the sputtered lubricating films are seldomly over 1 μm. In most applications they range from 2000 Å to 6000 Å. In special gyroscope applications a 700 Å thick film was reported to be the optimum thickness (2). Since these sputtered films are very thin when compared with the conventionally applied coatings, it is very critical to maintain proper sputtering conditions. These include, both surface cleaning and subsequent sputter deposition.

A layered solid lubricant such as MoS_2 or WS_2, has to meet two requirements in order to function as a lubricant: (1) it must exhibit intercrystalline slip, and (2) it must adhere to the surface. From an applications point of view, the real criterion of film quality is its performance. To optimize this performance in terms of low coefficient of friction, low wear, and long endurance lives it is essential to understand those factors which affect this performance.

The objective of this paper is to illustrate and describe by various electron microscopy and surface sensitive analytical techniques the interrelationships of film adherence, morphology, chemical composition, thickness, substrate temperature, topography, and chemistry relative to friction. Since sputtered MoS_2 is one of the most commonly used and investigated solid lubricants, its characterization and evaluation is of primary concern in this paper.

SPUTTERING AND ITS POTENTIALS

The growing interest of sputtering in tribological applications originates from the fact that virtually any solid lubricant can be directly (without a binder) applied in one operation on any bearing component, regardless of its geometry. Sputtering occurs by impact, rather than thermal evaporation and the technique is not limited by thermodynamic criteria. It offers great flexibility in forming coatings with graded interface composition, laminated layers, dispersion strengthened, and desirable additive additions. This technique allows one to tailor properties in ways not available with other techniques, due to the availability of the many sputtering modes and configurations. The type of potential applied can be varied, desirable target material can be fabricated into any possible composition and ratio (mixing various powders or constructing the target of several segments of different material), utilization of multiple targets either simultaneously or sequentially, reactive sputtering by introducing a desirable gas, use of auxiliary electrodes, utilization of magnetic fields, etc.

Of the many sputtering modes and arrangements which are used today, rf sputtering with a dc or rf bias is the most widely used technique for sputter deposition of solid lubricants. This mode of sputtering is shown photographically in Fig. 1. The apparatus has been described in detail in Ref. 4. The planar sputtering targets which were utilized were 12.7

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cm-diameter MoS_2, WS_2, and PTFE. The sputtering conditions for inorganic solid lubricants are generally in the range of 3.5 W/cm^2, for organics such as PTFE of 2 W/cm^2 at a frequency of 13.56 MHz and argon pressure of 2×10^{-2} torr. The distance from the target is maintained at approximately 2.5 cm. The component surfaces are cleaned by sputter etching prior to sputter deposition. Bearing components (cages and retainers), can be sputter coated without rotation, because the sputtered species leave the target at all possible angles as shown in Fig. 2. Most of the specimen surface is in direct line of sight with some portion of the target. Since the mean free path of the sputtered species is relatively short (<1 cm), the sputtered material will be scattered in random directions by collisions with particles in the plasma. Such scattering enables sputtered species to reach surfaces that are not in direct line of sight with the target. As a result, irregular, nonsymmetrical surfaces can be coated. When bearing balls are to be coated, they are placed in a screen pan (insert to fig. 1) and are kept in constant motion during the sputtering process in order to maintain uniform film coverage.

CHARACTERIZATION OF SPUTTERED MoS_2 FILMS

The degree of adherence and the mode of the morphological growth directly affect the quality of the sputtered film as a lubricant. Sputtered films grow in a complex plasma environment. The quality of the sputtered lubricant depends therefore on (1) sputter etching or biasing the substrate, (2) kinetic energy of the sputtered species, (3) plasma conditions, and (4) substrate temperature, chemistry, and topography. By evaluating the aforementioned factors, an understanding of the film performance, both desirable as well as deleterious effects during the lubrication cycle can be established.

Substrate Temperature and Film Morphology

Variation of the substrate temperature during MoS_2 sputtering results in changes in the nucleation and growth characteristics as well as in particle size. This temperature effect was determined by electron transmission micrographs and diffractograms. Various substrate temperatures from liquid nitrogen (-195° C) to 320° C were investigated (5). Temperature was found to affect film structure, at low temperatures the film was amorphous while at high temperature it was crystalline. Films with an amorphous diffraction pattern have frictional properties that are distinctly different from those of fine crystalline films. The interrelation between the substrate temperature, film morphology, grain size, and the coefficient of friction is summarized and represented in Fig. 3.

At the cryogenic temperatures the sputtered MoS_2 film (300 Å to 400 Å) exhibits a continuous nucleation sequence and has an amorphous diffraction pattern. When the film thickness of these amorphous films is increased to 2000 Å, and friction tested in vacuum, the coefficient of friction is high (0.4) and the films do not exhibit lubricating properties. They are very brittle and fragile.

MoS_2 films sputtered on substrates at ambient temperatures (25° C) and up to 320° C have distinct ridges (black lines) in the matrix as observed by electron transmission micrograph, and the diffractogram is characterized by sharp diffraction rings (fig. 3). The mean particle size increases from 50 Å to 110 Å as the substrate temperature is elevated from the ambient temperature of 25° C to 320° C.

The transition region from amorphous to crystalline has not been fully established, however at a substrate temperature of 7° C and below the formation of the dark ridges is suppressed. The electron diffraction patterns at the lower temperatures are more of the amorphous nature and the coefficient of friction is higher than observed for the films deposited between ambient temperatures (25° C) and 320° C. There is a clear trend in the transition region with the coefficient of friction decreasing from 0.4 to 0.04.

Similar morphological growth studies with sputtered MoS_2 in the 150° to 427° C range have been reported by Lavik and Campbell (6). It was revealed that the crystallites have a hexagonal structure, and there is considerable disorder in the film. The (ridges) black lines seen in the micrographs are crystals growing on edge with basal planes perpendicular to the film plane, whereas the gray areas are crystals with the basal planes parallel to the film plane. Lavik estimated that about 70 percent of the crystallites have a c-axis perpendicular to the film plane and 30 percent with the c-axis parallel to the film plane. In addition, slight rubbing of the film orients the platelets which were on edge and this is reflected in a color change from black to gray in the electron transmission micrographs.

When the thickness of the sputtered MoS_2 films at cryogenic and ambient temperatures is increased to several micrometers and the films are examined topographically and in cross section by scanning electron microscopy, distinct differences in film morphology are observed. Figure 4(a) is a photomicrograph of sputtered MoS_2 at ambient temperatures and it has the typical "feathery" lamellar structure whereas an MoS_2 sputtered at cryogenic substrate temperatures does not reveal the lamellar structure as indicated in Fig. 4(b). The cross sectional topographical morphologies of these two films are quite different and this is also reflected in the different friction coefficients observed with these films.

Surface Chemistry and Adherence

Sputtered MoS_2 films generally exhibit strong adherence not only to metallic surfaces but also to glass and polymer surfaces. Several exceptions have however been observed. These include copper, silver, and bronze surfaces where very poor adherence is noted and the film flakes (7). A micrograph (fig. 5) indicates the typical reaction islands during MoS_2 sputtering in the initial stages of film formation on copper. With continued sputtering the isolated reaction islands tend to combine by enlarging in size and finally a complete spalling of the film occurs as indicated in Fig. 6. Similar effects were also observed with silver and bronze surfaces.

Electron transmission micrographs and diffractograms were taken of 300 Å to 500 Å thick MoS_2 films sputtered on copper, silver, and bronze surfaces. Figure 7 presents a typical micrograph and diffractogram of sputtered MoS_2 on copper. The large white areas in the micrograph are voids where reaction products have been leached out during the dissolution of the copper substrate. The reaction products display a distinct color change from the usual gray to a dark blue-purple. The reaction products from copper and silver surfaces were analyzed by energy dispersive analysis. Figures 8(a) and (b) are the X-ray profiles of the reacted-flaked material on copper and silver surfaces, respectively. The X-ray peaks of Mo$L\alpha_1$ (2.293 keV) and S$K\alpha_{1,2}$ (2.307 keV) overlap in the spectrum and cannot be resolved. Therefore in Fig. 8 the most intense peaks are a combination of molybdenum and sulfur. However peaks at 8.04 keV are for copper $K\alpha$ and at 3.15 keV for silver $L\beta$. These results indicate that copper and silver have chemically reacted with the sputtered MoS_2 film. The crystalline size of the MoS_2 reaction products becomes noticeable, measuring between 100 Å to 200 Å and the corresponding diffractogram for MoS_2 on copper surfaces shows distinct sharp diffraction rings.

From the foregoing it can be concluded that a chemical reaction has occurred between sulfur and the metal (e.g., Cu or Ag) surface. It should be pointed out that the sputtered MoS_2 species are in a highly energetic state and are very prone to chemical adsorption and reaction. The energetic sputtered sulfur atoms which strike the substrate with a high velocity have a certain activation energy for reacting with the substrate as already shown by dispersive X-ray profiles. The selection of substrate material films becomes of paramount importance.

When the same copper, bronze, and silver surfaces were oxidized to form an oxide film about 1500 Å thick and subsequently sputtered with MoS_2, a strong adherent film was formed. Figure 9 shows the appearance of the flaked MoS_2 film on polished copper and bronze disks and highly adherent film on oxidized disks.

Topographical Effects

As the film thickness increases beyond 1/2 μm various unusual crystallographic defect growth features are formed in the matrix of the sputtered films. These defect features can arise from a variety of sources, the most common being surface topographical effects (microscratches, inhomogeneities, impurities, and contaminants) and imperfect crystalline film growth which are favorable nucleation sites. At these sites, an accelerated growth occurs relative to the matrix growth. As a consequence, the crystallographic defect features extend above the matrix surface. The most commonly observed defects are of the nodular type, they can grow individually, together, or overlap, forming complex aggregates, or they can be of an extreme or runaway growth type having a winding nature across the surface. Several of these defect structures in sputtered MoS_2 films are presented in the scanning electron micrographs of Figs. 10 to 13.

Nodules have a tendency to grow preferentially in high concentrations along the edge boundaries (fig. 10). Individual, overlapped or compounded nodules are shown in Figs. 11 and 12, and the extended or runaway types are presented in Fig. 13. In all these micrographs the pronounced "feathery" lamella-like structure is retained during the defect growth. The size of these defects increases as the thickness increases. A sputtered MoS_2 film about 2 μm thick can have compounded nodules over 10 μm in diameter and have the defects extend over 100 μm in length.

It is interesting to note that MoS_2 sputtered on substrates at cryogenic temperatures as shown in Fig. 14 have two distinct differences from films sputtered at ambient or elevated temperatures. At the cryogenic temperatures a high concentration per unit area of these nodules is formed and the defects seen in Fig. 14 do not have the lamella-like morphology but instead the film has a very densely packed structure.

These defects have undesirable effects on the film, since they have a tendency to be ejected from the matrix under stresses and strains as seen in Fig. 15, leaving a cavity. In precision type bearings of very close tolerances these defects have a tendency to break off and produce a higher volume of wear debris which may cause resistance to sliding and thus produce higher friction coefficients.

Substrate Biasing and Film Stoichiometry

The excellent lubricating properties of sputtered MoS_2 are attributed to the strong adherence. This adherence is directly dependent on the sputter etched surface and the energetics of the sputtered species. At low argon pressures, the sputtered material has enough energy to penetrate a few atomic layers into the substrate, which contributes to the strong adherence.

In sputtering technology it is very common to maintain a negative potential (bias) on the substrate during sputter deposition. This bias has a tendency to resputter the adsorbed or weakly adhered material, thus promoting film adherence. It has been established that the friction coefficient is very sensitive to the amount of bias applied to the substrate during MoS_2 sputter-deposition. The friction coefficient of the film increases steeply with a bias over -150 V dc and the lubricating properties of the film are completely lost at a bias of -350 V dc as indicated in the data in Fig. 16. This increase in friction is due to the compositional changes, resulting in depletion of sulfur in the film.

All MoS_2 sputter deposition has been therefore performed on sputter etched surfaces at ground potential. Under these conditions the stoichiometry of the film is maintained as analyzed by chemical techniques (4), and by Auger spectroscopy (8).

Atmospheric Effects

It is well documented in the literature that the coefficient of friction for MoS_2 increases with the partial pressure of water vapor in the air (9). The effects of atmospheric pressure on the friction coefficient for sputtered MoS_2 films have been evaluated. The changes in the friction coefficient with changes in pressure from atmospheric pressure (760 torr) and relative humidity of 60 percent to 10^{-9} torr is presented in the data of Fig. 17. These data were obtained in an ultrahigh-vacuum friction apparatus during sliding. The friction curve in Fig. 17 was constructed by starting the friction test at atmospheric conditions and gradually evacuating the chamber and monitoring the change in friction. The reverse procedure was also performed wherein the friction test was started at 10^{-9} torr and the pressure was gradually increased until atmospheric conditions were reached. The resulting friction curves had very close agreement in their profile, regardless how it was determined, either by evacuation or pressurizing the chamber. A continuous change occurred in the friction coefficient between 100 and 400 torr. Above 400 torr the friction coefficient was steady at about 0.15, below 100 torr it stabilized at 0.04. Friction tests of sputtered MoS_2 films were also conducted in dry air, nitrogen, and argon. The friction coefficient under these conditions was the same, namely 0.04.

Duplex Sputtered Films

Angular contact, 204-size, 440C stainless steel bearings (races and cages) were sputter coated with a 1000 Å thick underlayer of Cr_3Si_2, and subsequently sputtered with MoS_2 film about 6000 Å. Another series of the same bearings were directly sputter coated with only a 6000 Å thick MoS_2 film. These two sets of bearings were tested for film endurance at speeds of 1750 rpm, thrust load of 137.9 newtons in high vacuum (10). Figure 18 presents a comparison of the endurance lives of the duplex films and the MoS_2 films. The endurance life with the Cr_3Si_2 underlayer was over 1000 hours, compared to 187 hours for the directly sputtered MoS_2 films.

OTHER SPUTTERED SOLID LUBRICANTS

Laminar Solids

In addition to MoS_2 the other laminar (MX_2) compounds having lubricating properties have not as yet attracted any great interest for deposition by sputtering. However very limited investigations have been performed with WS_2 and $NbSe_2$ sputtered films. Sputtered WS_2 films when tested during sliding conditions in vacuum exhibit similar friction and wear characteristics to sputtered MoS_2. Typical wear tracks of sputtered WS_2 film are shown in Fig. 19. When these films are friction tested in vacuum the friction coefficient stabilizes at 0.05 and endurance lives are over million cycles. When the same film was tested under identical experimental conditions in atmosphere the coefficient of friction immediately increased to 0.22 and film failure occurred after only 1500 cycles. This premature failure of the film under atmospheric conditions is explained on the basis of H_2SO_4 formation as determined by the discoloration of litmus paper, and the corrosive effect on the surface.

Soft Metals

Soft metals such as gold, silver, and lead have been sputter-deposited for tribological applications. However, it would appear more desirable wherever possible, that these soft metals should be applied by ion plating. This technique has the ability to form graded interfaces which contributes to excellent adherence.

Organic Polymers

Of the many organic compounds available for tribological applications only teflon (PTFE) polytetrafluoroethylene and the polyimides have been sputtered (11). In recent years increased interest in rf sputtered thin PTFE films has developed in electronics for insulation and encapsulation as well as for capacitor dielectrics (12). In tribology, preliminary results with sputtered PTFE films indicate that the coefficient of friction is comparable to that of the original PTFE, which may vary from 0.08 to 0.2 (13). The coefficient of friction for PTFE depends on the load, sliding speed, and film thickness. When the thickness of the sputtered film dominates the frictional behavior, the coefficient of friction decreases markedly with increase in load for any given thickness (13).

Sputtered PTFE films display excellent adherence and uniformity not only to metal but also to glass, wood, and paper surfaces. All these rf sputtered PTFE films exhibit a yellowish color in appearance, which is common to all polymers prepared in a glow discharge and is probably due to disordering in the polymer structure. As the sputtering rates are increased a darkening in color occurs and the chemical composition of the film changes with depletion in carbon content (11). Electron transmission micrographs and diffractograms of sputtered PTFE films show an amorphous type of structure. These thin films exhibit high packing density, without any micro-porosity. It is believed that the sputtered amorphous film has a cross-linked structure with a radically shorter chain length.

A unique medical application now exists where sputtering is the only means by which a thin 1000 Å thick PTFE film can be applied to surgical needles in order to improve the technique for cataract operations. The components (needles and housing) for a tiny cutter pump used in cataract operations was coated with a thin uniform film of PTFE. The coated needles are presented in the photograph in Fig. 20. The purpose of the sputtered PTFE film was to act as a lubricant to reduce the insertion and removal of the pump through a small incision in the cornea of the eye.

CONCLUDING REMARKS

Thin, adherent and uniform films, essentially of any solid lubricant can be applied to sliding or rolling surfaces in one operation, regardless of geometry, by sputtering techniques. Sputter deposition can produce many modes and configurations such as duplex films or films impregnated with additives.

Of the many solid lubricants sputtered, MoS_2 has been most widely investigated and is already finding an increased use for commercial applications. Application of PTFE (teflon) by sputtering results in very strong adherence and the films show great promise.

It was shown by electron microscopy and surface sensitive analytical techniques that the lubricating properties of the sputtered MoS_2 films are directly affected by the sputtering parameters selected (power density, sputter etching, dc-biasing, etc.) and the substrate temperature, chemistry, topography and the environmental conditions during the friction test. The electron micrographs and diffractograms of sputtered MoS_2 films clearly illustrate the resultant changes in film morphology which influence the film adherence and frictional properties. These thin sputtered films will perform reliably only when the proper sputtering parameters and substrate conditions are selected.

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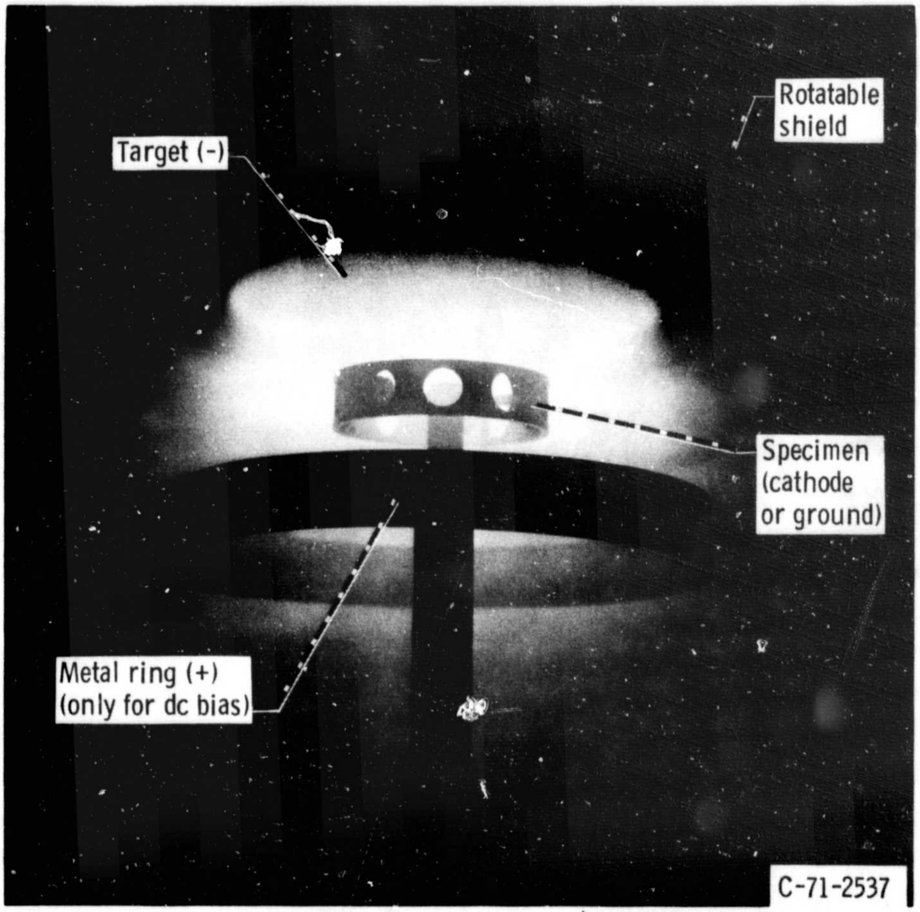

Figure 1. - Radiofrequency diode sputtering apparatus with direct-current bias.

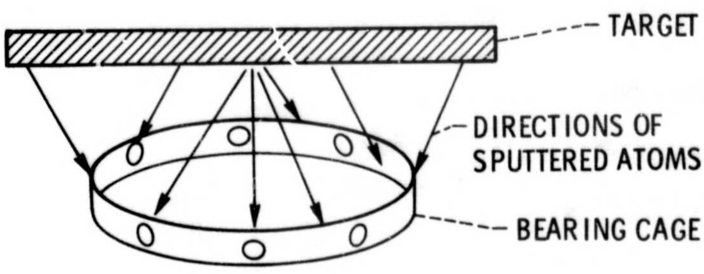

Figure 2. - Schematic of sputter coating complex surfaces.

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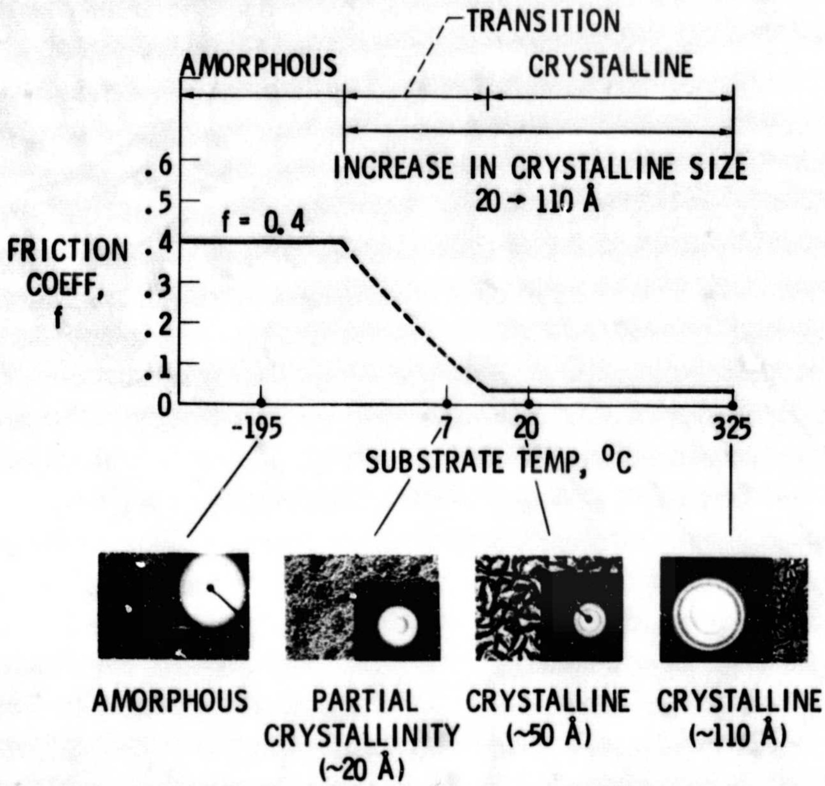

Figure 3. - Substrate temperature effects on MoS_2 film morphology and friction coefficient.

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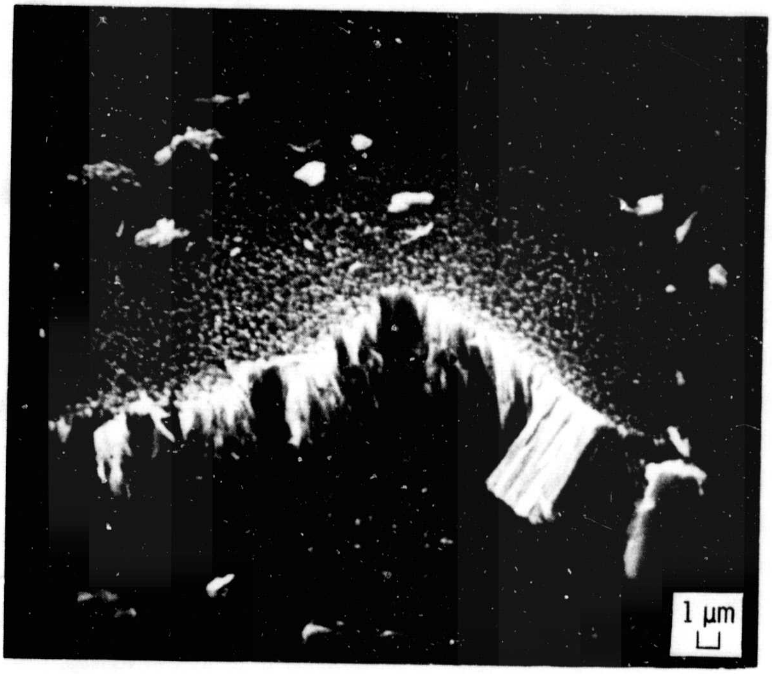
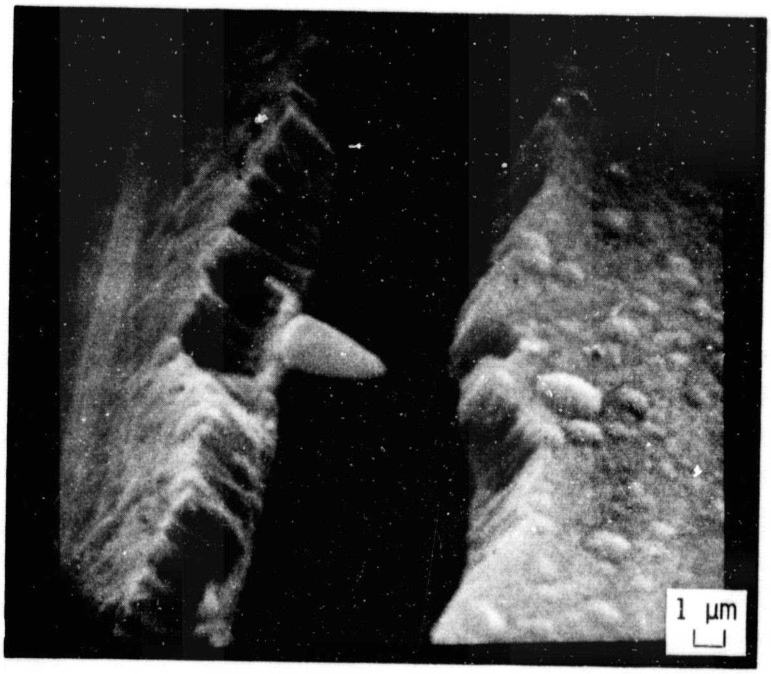


(a) AT AMBIENT TEMPERATURE.



(b) AT CRYOGENIC TEMPERATURE (-195° C).

Figure 4. - Surface and cross sectional structure of sputtered MoS_2 on steel.

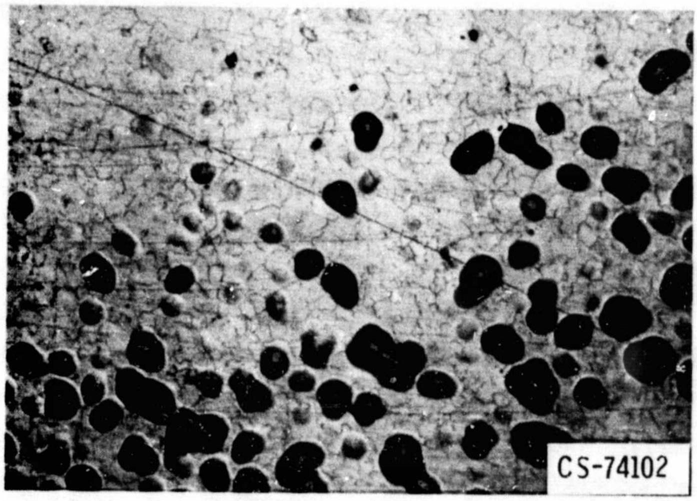


Figure 5. - Sputtered molybdenum disulfide on polycrystalline copper in the initial state of film formation. X80.

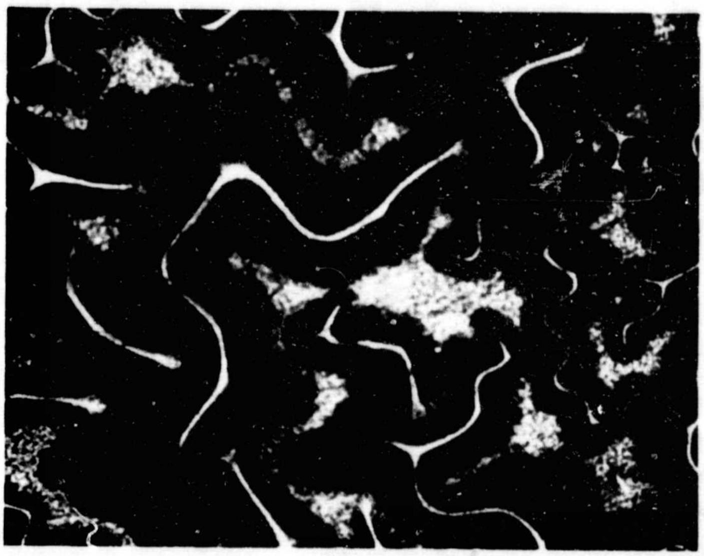


Figure 6. - Sputtered molybdenum disulfide on polycrystalline copper in the final state of film formation, with blistering. X40.

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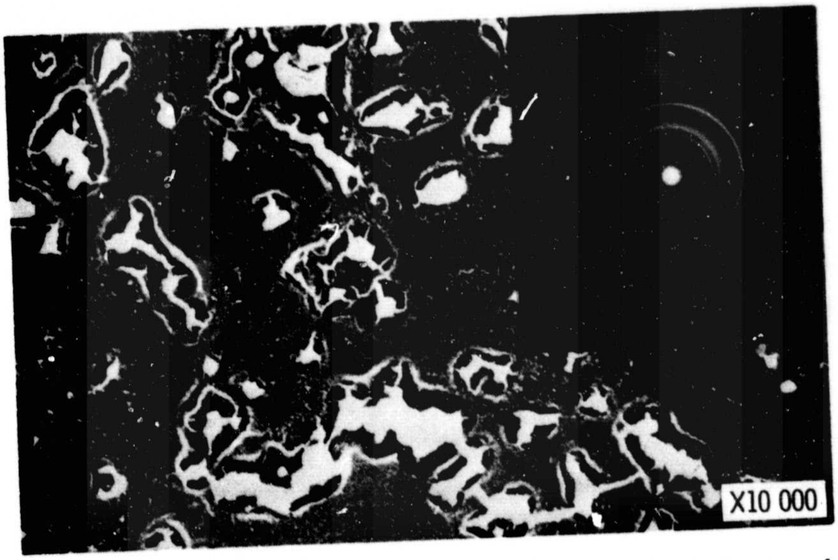
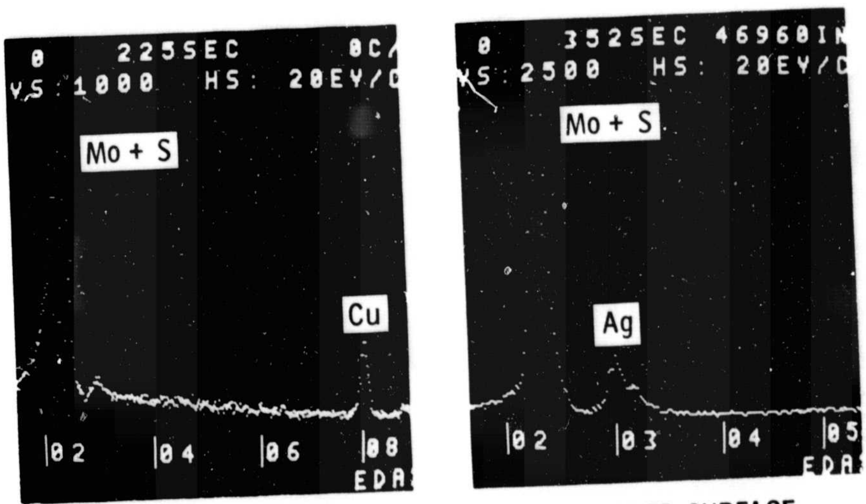
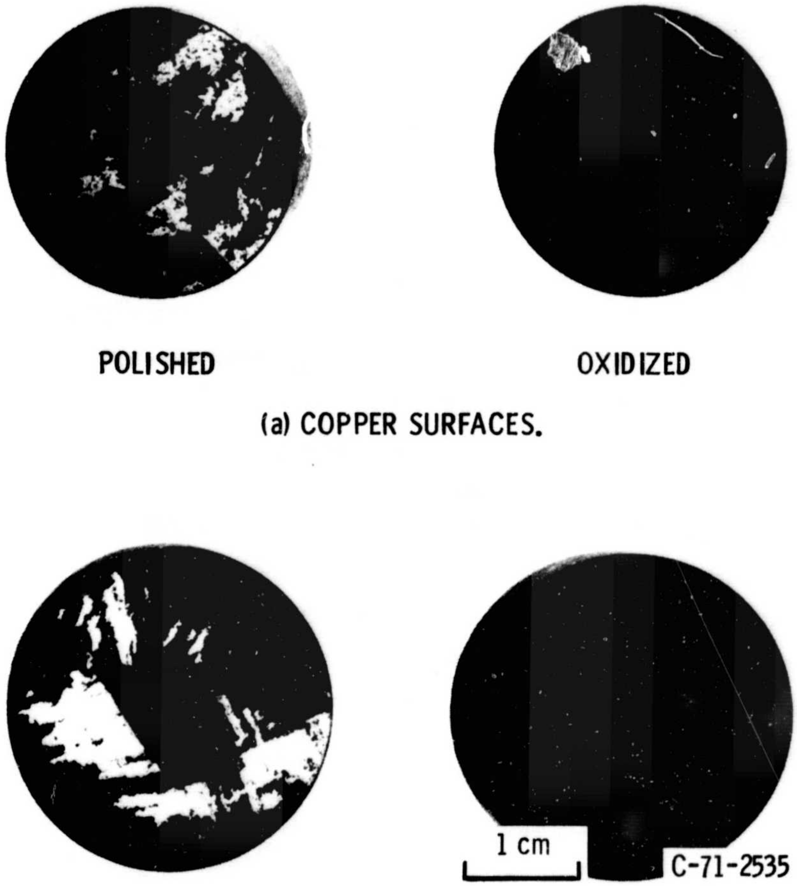


Figure 7. - Electron transmission micrograph and diffractogram of sputtered molybdenum disulfide on copper.



ON COPPER SURFACE ON SILVER SURFACE

Figure 8. - Energy dispersive x-ray profiles of the reacted, flaked sputtered MoS_2 film.



POLISHED OXIDIZED

(a) COPPER SURFACES.

POLISHED OXIDIZED

(b) BRONZE SURFACES.

Figure 9. - Sputtered molybdenum disulfide film on copper and bronze surfaces.

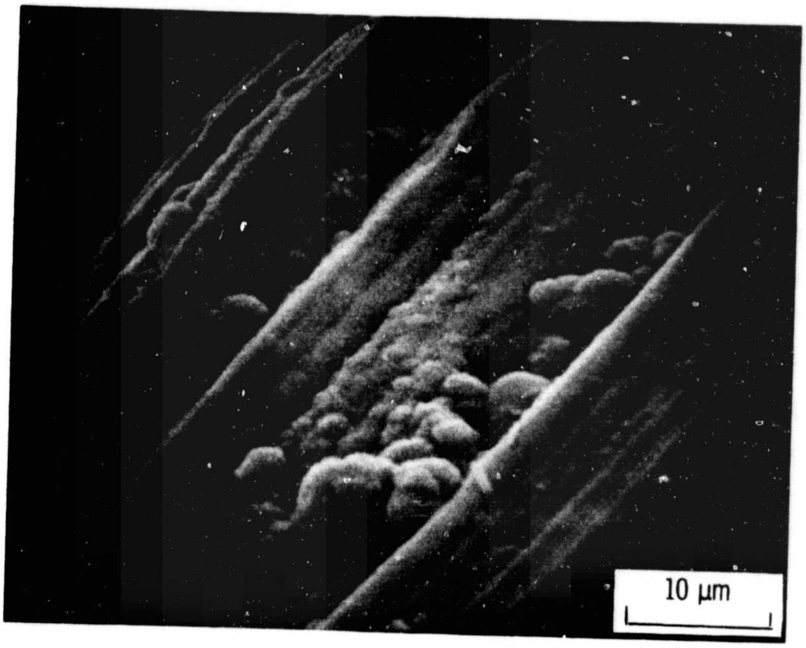


Figure 10. - Surface of sputtered MoS_2 on 304 stainless steel sanded to 22.5×10^{-2} μm finish.

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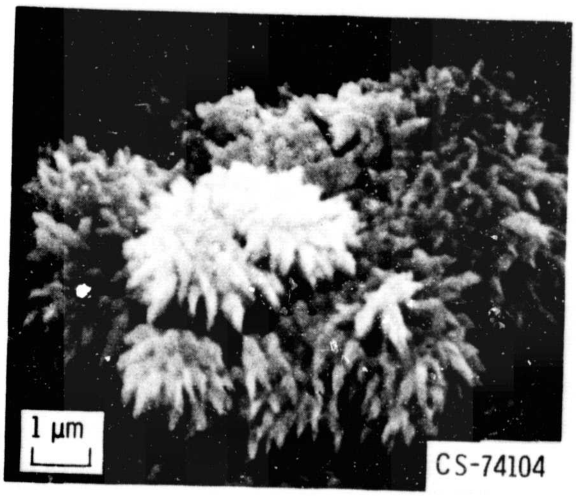

Figure 11. - Surface structure of a nodule of sputtered MoS_2 on 304 stainless steel with 5×10^{-2} μm finish.

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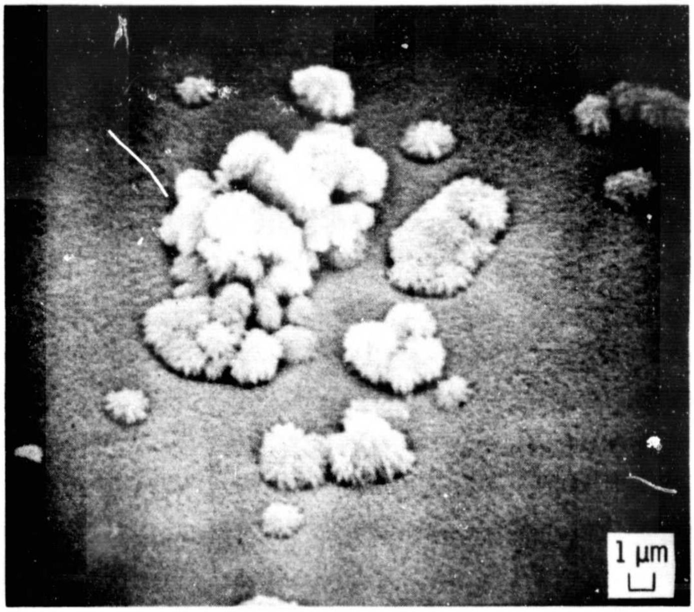

Figure 12. - Surface structure of sputtered MoS_2 on 304 stainless steel with $5x10^{-2}$ μm finish.

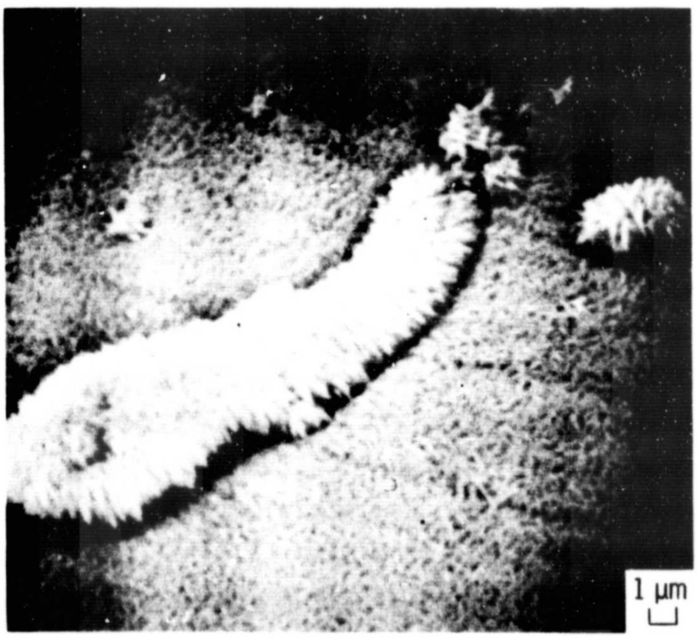

Figure 13. - Longitudinal surface outgrowths in sputtered MoS_2 on steel.

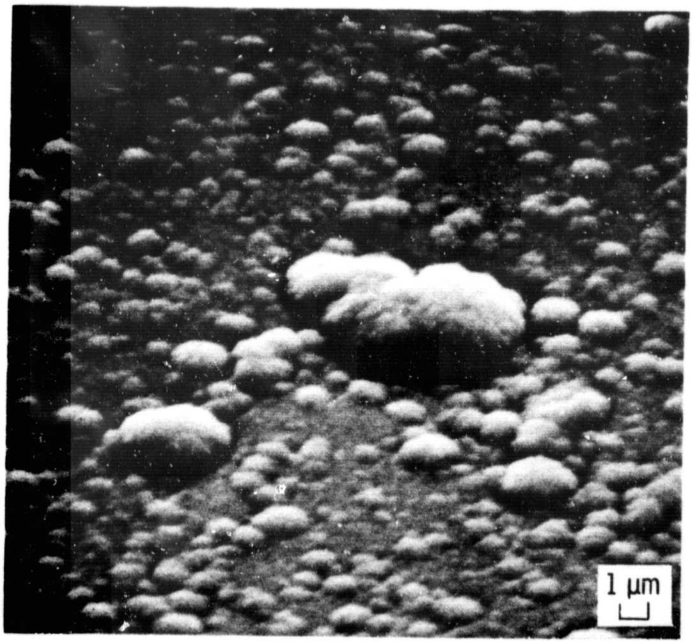

Figure 14. - Surface structure of sputtered MoS_2 on glass at cryogenic temperatures.

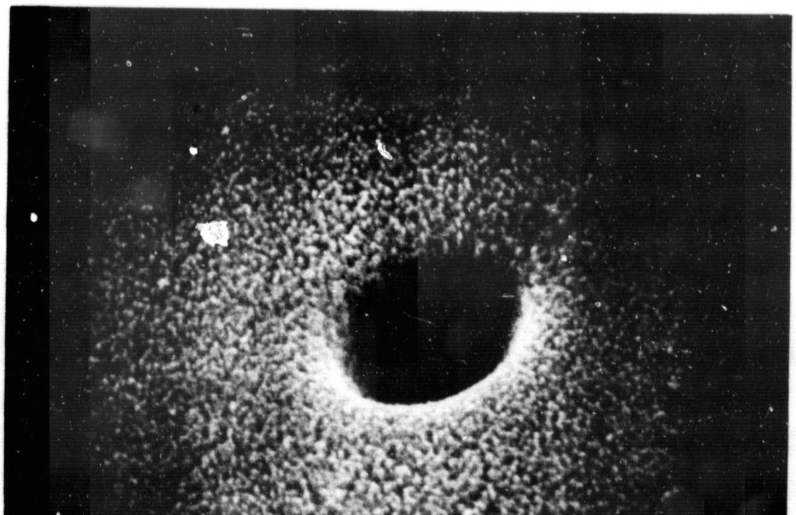

Figure 15. - Cavity on sputtered MoS_2 left by ejected nodule.

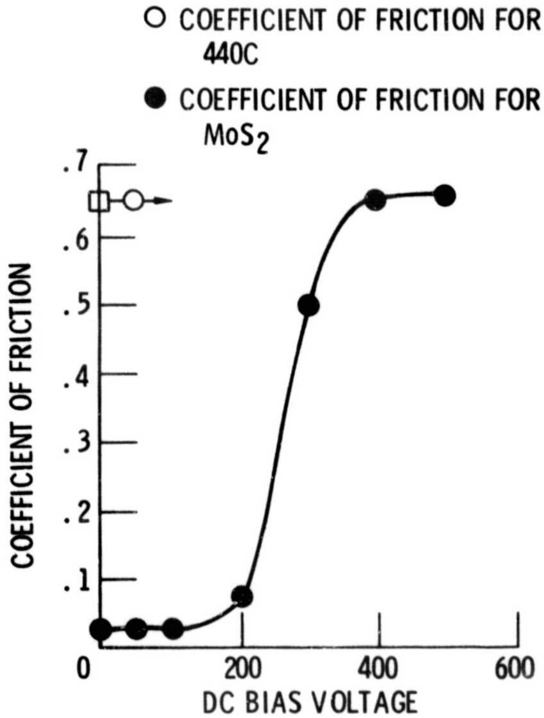

Figure 16. - Effect of negative DC bias on coefficient of sliding friction for sputtered MoS_2. Load, 250 grams; speed, 40 rpm; substrate/rider, 440C/440C at 1×10^{-3} torr.

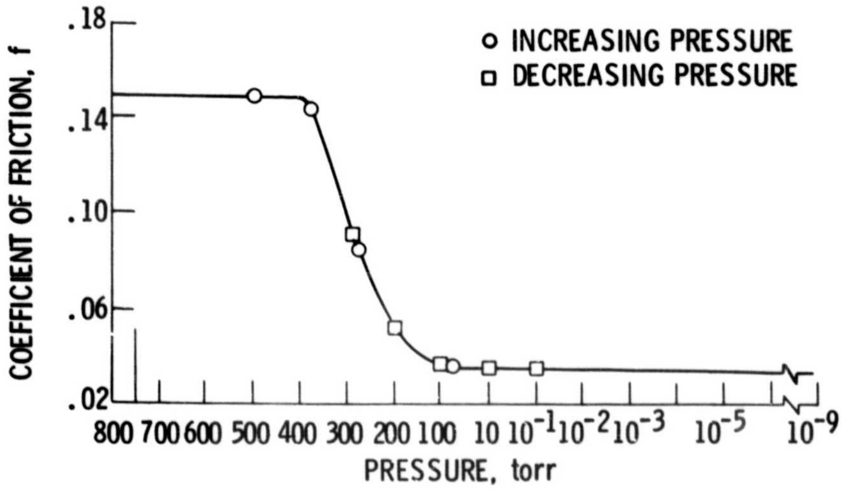

Figure 17. - Effect of pressure on coefficient of sliding friction for sputtered MoS_2. Load, 250 grams; speed, 40 rpm; substrate/rider, Ni/Ni; room temperature.

E-9650

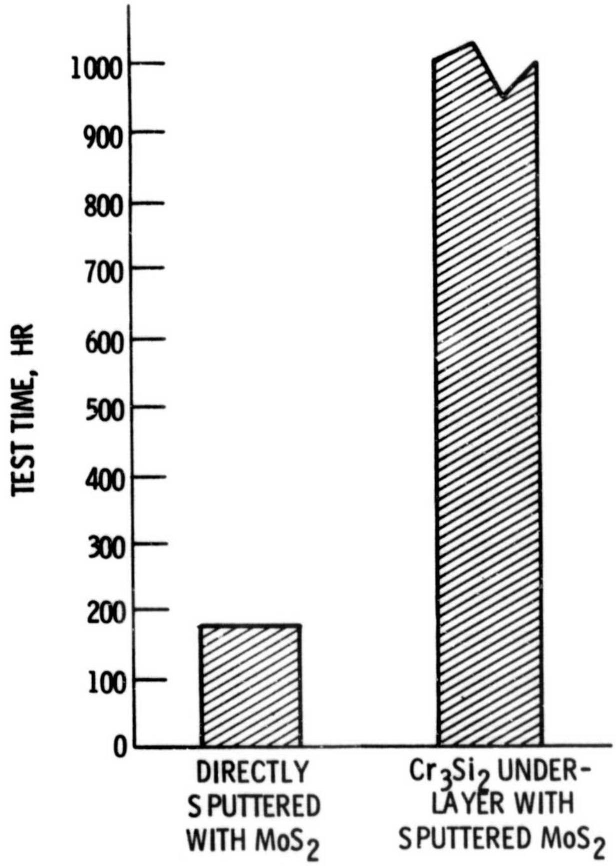

Figure 18. - Endurance lives of 440C stainless-steel ball bearings with sputtered MoS_2 films on races and cage - with and without a Cr_3Si_2 underlayer.

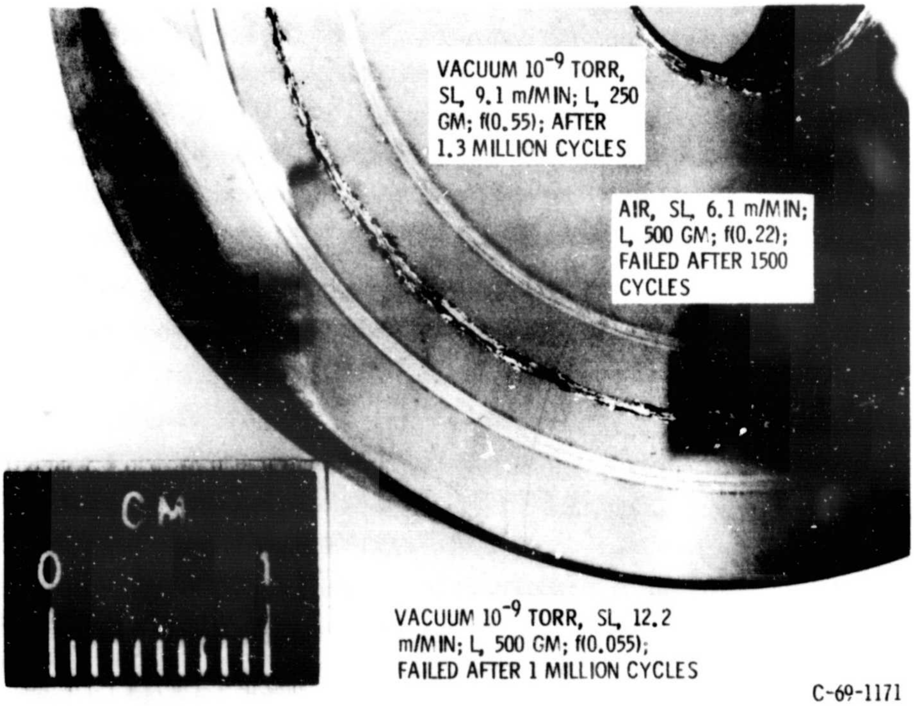

Figure 19. - Wear tracks on a (Ni-Cr) disk with sputtered WS_2 film (~2500 A) after sliding against a 4.75 mm hemispherical nickel rider (SL, sliding velocity; L, load).

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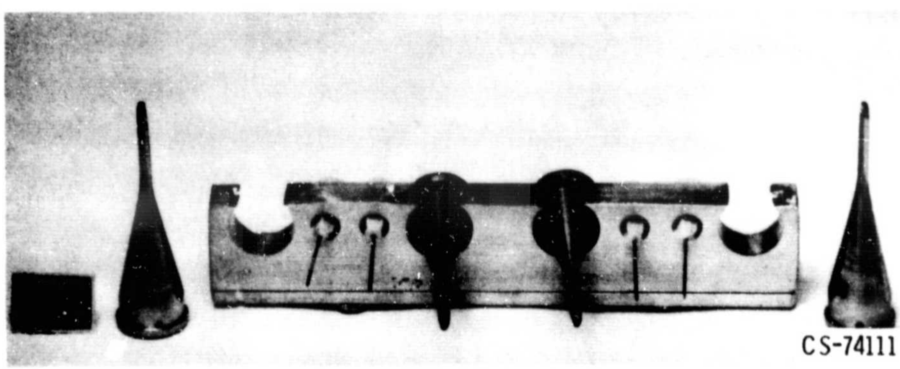

Figure 20. - Hypodermic needles and protective housings with sputtered teflon.