EVALUATION OF SELECTED SOLID LUBRICATING FILMS

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ABSTRACT

An investigation was conducted to examine the friction and wear properties of bonded molybdenum disulfide (MoS_2), magnetron-sputtered MoS_2, ion-plated silver, ion-plated lead, magnetron-sputtered diamondlike carbon (MS DLC), and plasma-assisted, chemical-vapor-deposited DLC (PACVD DLC) films in sliding contact with 6-mm-diameter AISI 440C stainless steel balls. Unidirectional ball-on-disk sliding friction experiments were conducted with a load of 5.9 N and a sliding velocity of 0.2 m/s at room temperature in three environments: ultrahigh vacuum (vacuum pressure, 7×10^{-7} Pa), humid air (relative humidity, ~20 percent), and dry nitrogen (relative humidity, <1 percent). The main criteria for judging the performance of the solid lubricating films were coefficient of friction and wear rate, which had to be less than 0.3 and on the order of 10^{-6} $mm^3/N{\cdot}m$ or less, respectively. The bonded MoS_2 and magnetron-sputtered MoS_2 films met the criteria in all three environments. The ion-plated lead and silver films met the criteria only in ultrahigh vacuum but failed in humid air and in dry nitrogen. The MS DLC and PACVD DLC films met the requirements in humid air and dry nitrogen but failed in ultrahigh vacuum.

Keywords: MoS_2 films; Lead films; Silver films; DLC films

INTRODUCTION

Compiling manufacturers' standard test results for a number of lubricant formulations can aid mechanism design engineers (i.e., users) in selecting the best lubricant for an application. However, such data can at best only narrow the field to a specific class of lubricants. Deciding on the optimum lubricant formulation for a specific application requires more custom-design, element, component, and full-scale testing. However, after the optimum lubricant has been chosen, such standard tests can also be useful for quality control. This is especially important during a long space program, where satellites or launch vehicles are built over a period of years during which lubricant formulations and film application procedures might undergo change. For solid lubricant films the end user should request that standard test coupons be coated along with the actual parts. Testing of each lubricant batch will ensure that the manufacturing quality remains constant throughout the life of the program.

The technology of solid lubrication has advanced rapidly in the past four decades, conforming primarily to the needs of the automobile and aerospace industries [1–12]. Solid lubrication can be accomplished in several modes: lubricating powders, bonded films, lubricant composites (metal and plastic based), and lubricating coatings and films. In this study, however, we chose primarily commercially developed, bonded films and deposited coatings and films. This paper is limited to evaluating the friction and wear properties of the solid lubricating films selected.

SELECTED SOLID LUBRICATING FILMS

We selected six types of commercially developed, affordable dry solid film lubricants as shown in table 1:

1. Bonded molybdenum disulfide (MoS_2), the most widely used mode
2. Magnetron-sputtered MoS_2

3. Ion-plated silver
4. Ion-plated lead
5. Magnetron-sputtered diamondlike carbon (MS DLC)
6. Plasma-assisted, chemical-vapor-deposited diamondlike carbon (PACVD DLC)

Some earlier work on these films was reported in references [13–15].

Molybdenum Disulfide Films

Molybdenum disulfide is a versatile and useful solid lubricant [1–3]. Bonded films are much more readily available than sputtered films, but sputtered films are easy to incorporate in precision bearing systems. In this study we selected both bonded and magnetron-sputtered MoS_2 films. As shown in table 1, the bonded MoS_2 films were relatively rough, having centerline-average roughness R_a of 1.2 µm, and the magnetron-sputtered MoS_2 films were relatively smooth, having R_a in the range of 32 nm. The bonded films were 10 times thicker than the sputtered films.

Silver and Lead Films

Soft metal films can be deposited as lubricating films on harder substrates by conventional electroplating or by physical vapor deposition methods, such as evaporation, vacuum sputtering, and ion plating. Ion plating and vacuum sputtering permit close control of film deposition and thickness and can provide good adhesion to the substrate [5]. In this study we selected ion-plated silver and ion-plated lead films [13, 14]. These films were relatively smooth, having R_a of 30 and 98 nm, respectively, and were approximately 0.5 µm thick and uniform.

Diamondlike Carbon Films

A new category of solid lubricants and lubricating films, diamond and related hard materials, is growing. Particularly, commercial interest in mechanical parts and components coated with diamondlike carbon (DLC) is continuously expanding. The cost is affordable and generally similar to that of carbide or nitride films deposited by chemical or physical vapor deposition (CVD and PVD) techniques. The surface smoothness, high hardness, low coefficient of friction, low wear rate, and chemical inertness of DLC coatings and films, along with little restriction of geometry and size, make them well suited for applications as solid lubricants combating wear and friction.

We selected magnetron-sputtered (MS) DLC and plasma-assisted (PA) CVD DLC films. These films were relatively smooth, having R_a of 43 and 29 nm, respectively, and were approximately 2 to 5 μm thick and uniform. The MS DLC films had a multilayer structure and were prepared by using two chromium targets, six tungsten carbide (WC) targets, and methane (CH_4) gas. The multilayer film comprised 20- to 50-nm-thick, alternating layers of WC and carbon. The Vickers hardness number was approximately 1000. The PACVD DLC films were prepared by using radiofrequency plasma and consisted of two layers, an approximately 2-μm-thick DLC top layer on an approximately 2-μm-thick silicon-DLC underlayer. The DLC top layer was deposited by using CH_4 gas at a total pressure of 8 Pa with a power of 1800 to 2000 W at –750 to –850 V for 120 min. The silicon-containing DLC underlayer was deposited by using a mixture of CH_4 and tetramethylsilane ($C_4H_{12}Si$) gases. The ratio of the concentration of CH_4 and $C_4H_{12}Si$ used was 90:18 (std cm^3/min) at a total pressure of 10 Pa with a power of 1800 to 2000 W at –850 to –880 V for 60 min. The Vickers hardness number was 1600 to 1800.

EXPERIMENTAL PROCEDURE

We examined the friction and wear properties of the selected solid lubricating films in ultrahigh vacuum, in humid air at a relative humidity of approximately 20 percent, and in dry nitrogen at a relative humidity of less than 1 percent. Unidirectional pin-on-disk sliding friction experiments were conducted with 6-mm-diameter American Iron and Steel Institute (AISI) 440C stainless steel balls in sliding contact with the solid lubricating films at room temperature in each environment at the conditions shown in table 2. The 440C balls (grade 10) used were smooth, having R_a of 6.8 nm with a standard deviation of 1.8 nm. The resultant solid lubricating films and their wear surfaces were characterized by scanning electron microscopy (SEM), energy-dispersive x-ray spectroscopy (EDX), and surface profilometry. SEM and EDX were used to determine the morphology and elemental composition of wear surfaces and wear debris. The sampling depth of EDX for elemental information ranged between 0.5 and 1 μm in this study. Surface profilometry was used to determine the surface morphology, roughness, and wear of the films.

EXPERIMENTAL RESULTS AND DISCUSSION

Comparison of Steady-State Coefficients of Friction and Wear Rates

Table 3 and figures 1 to 3 summarize the steady-state coefficients of friction, the film wear rates (dimensional wear coefficients), and the ball wear rates after sliding contact in all three environments: ultrahigh vacuum, humid air, and dry nitrogen. The data presented in the table and figures reveal the marked differences in friction and wear resulting from the environmental conditions and the solid lubricating film materials.

Ultrahigh vacuum.—As shown in figure 1, in sliding contact with 440C stainless steel balls in ultrahigh vacuum, the bonded MoS_2 films had the lowest coefficient of friction, lowest film wear rate, and lowest ball wear rate. The MS MoS_2 films also had low coefficient of friction, low film wear rate, and low ball wear rate, similar to those for the bonded MoS_2. The film and ball wear rates in ultrahigh vacuum were in ascending order: bonded MoS_2 < MS MoS_2 < ion-plated silver < ion-plated lead < PACVD DLC < MS DLC. The coefficients of friction followed a similar ascending order. The MS DLC films had the highest coefficient of friction, the highest film wear rate, and the highest ball wear rate in ultrahigh vacuum.

Humid air.—As shown in figure 2, in sliding contact with 440C stainless steel balls in humid air, the PACVD DLC film had the lowest coefficient of friction, lowest film wear rate, and lowest ball wear. The bonded MoS_2, MS MoS_2, and MS DLC films generally had low coefficients of friction, low film wear rates, and low ball wear rates, similar to those for the PACVD DLC. The wear rates of the solid lubricating films in humid air were in ascending order: PACVD DLC < MS DLC < MS MoS_2 < bonded MoS_2 < ion-plated lead < ion-plated silver. The coefficients of friction and wear rates of the balls followed a similar ascending order. The ion-plated silver films had the highest coefficient of friction, highest film wear rate, and highest ball wear rate in humid air.

Dry nitrogen.—As shown in figure 3, in sliding contact with 440C stainless steel balls in dry nitrogen, the MS MoS_2 films had the lowest coefficient of friction. The bonded MoS_2, MS MoS_2, and PACVD DLC films had low coefficients of friction, low film wear rates, and low ball wear rates. However, the ion-plated silver and ion-plated lead films had high friction and high wear. The wear rates of the solid lubricating films in dry nitrogen were in ascending order: PACVD DLC < MS MoS_2 < bonded MoS_2 < MS DLC < ion-plated lead < ion-plated silver. The coefficients of friction and wear rates of the balls followed a similar ascending order.

Wear Life (Endurance Life)

The sliding wear (endurance) life of the solid lubricating films deposited on 440C stainless steel disks was determined to be the number of passes at which the coefficient of friction rose to 0.30 in a given environment. The sliding wear lives, presented in figures 4 to 6 and table 3, varied with the environment and the type of solid lubricating film.

Ultrahigh vacuum.—As shown in figure 4, in sliding contact with 440C stainless steel balls in ultrahigh vacuum, the bonded MoS_2 films had the longest wear life, over 1 million passes. The wear lives of the solid lubricating films in ultrahigh vacuum were in descending order: bonded MoS_2 > ion-plated silver > MS MoS_2 > ion-plated lead > PACVD DLC > MS DLC.

Humid air.—As shown in figure 5, in sliding contact with 440C stainless steel balls in humid air, both PACVD DLC and MS DLC had the longest wear lives, over 300 000 passes. The wear lives of the solid lubricating films in humid air were in descending order: PACVD DLC > MS DLC > MS MoS_2 > bonded MoS_2 > ion-plated lead > ion-plated silver.

Dry nitrogen.—As shown in figure 6, in sliding contact with 440C stainless steel balls in dry nitrogen, the bonded MoS_2, MS MoS_2, and PACVD DLC films had the longest wear lives, over 1 million passes. The wear lives of the solid lubricating films in dry nitrogen were in descending order: bonded MoS_2 > MS MoS_2 > PACVD DLC > MS DLC > ion-plated lead > ion-plated silver.

Sliding Wear Behavior, Wear Debris, and Transferred Wear Fragments

Adhesion and plastic deformation played important roles in the friction and sliding wear of the selected solid lubricating films in sliding contact with the 440C stainless steel balls in ultrahigh vacuum, humid air, and dry nitrogen [13–15]. The worn surfaces of both the films and the balls contained wear debris particles. Examination of the surface morphology and compositions of the worn surfaces by SEM and EDX provided detailed information about plastic deformation of the solid lubricating films, wear debris, and transferred wear fragments produced during sliding in all three environments. Marked plastic deformation occurred in the six solid lubricating films. Smeared, agglomerated wear debris accumulated around the contact borders, particularly at the rear end of the wear scars. All sliding involved adhesive transfer of materials.

Bonded MoS_2.—The 440C stainless steel balls left transferred steel wear fragments in the wear tracks on the bonded MoS_2 films in all three environments [13]. During sliding the relatively coarse asperities of the bonded MoS_2 films were deformed plastically, and the tips of the asperities were flattened under load. The ball wear scars contained transferred MoS_2 wear fragments. Wear fragments of MoS_2 and steel usually adhered to the counterpart surface or came off in loose form. Another form of adhesive MoS_2 transfer was found in sliding wear. SEM and EDX showed that a thin MoS_2 layer (or sheet) was generated over the entire ball wear scars in all three environments.

Magnetron-sputtered MoS_2.—The 440C stainless steel balls left transferred steel wear fragments in the wear tracks on the MS MoS_2 films in all three environments [13]. The fine asperities of the sputtered MoS_2 films were flattened and elongated in the sliding direction by plastic deformation, revealing a burnished appearance. The ball wear scars contained transferred MoS_2 wear fragments. A thin MoS_2 layer was also generated over the entire ball wear scars.

According to the elemental concentrations, in ultrahigh vacuum much less transfer occurred between the films and the balls and vice versa with MS MoS_2 than with bonded MoS_2. In both humid air and dry nitrogen, however, more stainless steel transferred to the sputtered MoS_2 films than to the bonded MoS_2 films, although much less bonded MoS_2 transferred to the steel. A thin MoS_2 layer was also generated over the entire ball wear scars in humid air and dry nitrogen.

Ion-plated silver.—The 440C stainless steel balls left a small amount of transferred steel wear fragments in the wear tracks on the ion-plated silver films in all three environments [13]. The fine asperities of the ion-plated silver films were flattened and elongated in the sliding direction by plastic deformation, revealing a burnished appearance. Severe plastic deformation and shearing occurred in the silver films during sliding.

According to the elemental concentrations, after sliding in ultrahigh vacuum the entire ball wear scar contained thick transferred layers (or sheets) of silver, and plate-like silver debris particles were deposited at the edges of the film wear track. In contrast, after sliding in humid air the ball wear scar contained an extremely small amount of transferred silver particles. This result suggests that oxidation of silver during sliding in humid air may prevent large silver transfer. However, plate-like silver wear debris was deposited at the edges of the film wear track in humid air. After sliding in dry nitrogen the ball wear scar contained transferred silver plates and particles, and plate-like silver wear debris was deposited at the edges of the film wear track. Severe plastic deformation and shearing occurred in the silver film during sliding in dry nitrogen.

Ion-plated lead.—The 440C stainless steel balls left a small amount of transferred steel wear fragments in the wear tracks on the ion-plated lead films in all three environments [14]. The fine asperities of the ion-plated lead films were flattened and elongated in the sliding direction by plastic deformation, revealing a burnished appearance. Severe plastic deformation and shearing occurred in the lead film during sliding.

According to the elemental concentrations, after sliding in ultrahigh vacuum the entire ball wear scar contained thick transferred layers (or sheets) of lead. Plate-like lead debris was found at the edges of the film wear track. In contrast, after sliding in humid air the ball wear scar contained an extremely small amount of transferred lead debris particles. This result suggests that oxidation of lead, like silver oxidation, during sliding in humid air may prevent large lead transfer. However, plate-like lead wear debris was deposited at the edges of the film wear track in humid air. After sliding in dry nitrogen the ball wear scar contained transferred lead plates and particles, and plate-like lead wear debris was deposited at the edges of the film wear track.

Magnetron-sputtered DLC.—With MS DLC films sliding involved generation of fine wear debris particles and agglomerated wear debris and transfer of the worn materials in all three environments [15].

According to the elemental concentrations, after sliding in ultrahigh vacuum the 440C stainless steel ball left a roughened worn surface and a small amount of transferred steel fragments in the wear track on the MS DLC film. The ball wear scar contained fine steel particles and a small amount of transferred DLC fragments. The wear mechanism was that of small fragments chipping off the DLC surface.

After sliding in humid air the 440C stainless steel ball left a small amount of transferred steel fragments in the wear track on the MS DLC film. The fine asperities of the MS DLC film were flattened and elongated in the sliding direction by plastic deformation, revealing a burnished appearance. The entire ball wear scar contained transferred patches and thick transferred layers (or sheets) of MS DLC. Plate-like DLC wear debris was also deposited at the edges of the wear scar. Severe plastic deformation and shearing occurred in the DLC film during sliding in humid air.

After sliding in dry nitrogen the 440C stainless steel ball left an extremely small amount of transferred steel wear debris in the wear track on the MS DLC film. In addition, smeared, agglomerated DLC wear debris was deposited on the film. The fine asperities of the MS DLC film were flattened and elongated in the sliding direction by plastic deformation, revealing a burnished appearance. The ball wear scar contained transferred DLC wear debris.

Plasma-assisted, chemical-vapor-deposited DLC.—With PACVD DLC films, like MS DLC films, sliding involved generation of fine wear debris particles and agglomerated wear debris and transfer of the worn materials in all three environments [15].

According to the elemental concentrations, after sliding in ultrahigh vacuum the 440C stainless steel ball left smeared, agglomerated DLC wear debris and a small amount of transferred steel fragments in the film wear track. The ball wear scar contained fine steel particles and large smeared, agglomerated patches containing transferred DLC fragments. The wear mechanism was adhesive, and plastic deformation played a role in the burnished appearance of the smeared, agglomerated wear debris.

After sliding in humid air the 440C stainless steel ball left a small amount of transferred steel fragments in the film wear track. The fine asperities of the PACVD DLC film were flattened and elongated in the sliding direction by plastic deformation, revealing a burnished appearance. The smooth ball wear scar contained an extremely small amount of transferred DLC wear debris.

After sliding in dry nitrogen the 440C stainless steel ball left DLC wear debris, micro-pits, and an extremely small amount of transferred steel wear debris in the wear track on the PACVD DLC film. The fine asperities of the film were flattened and elongated in the sliding direction by plastic deformation, revealing a burnished appearance. The ball wear scar contained fine grooves in the sliding direction, steel wear debris, and a small amount of transferred DLC wear debris.

SUMMARY OF REMARKS

To evaluate recently developed, commercially available, dry solid film lubricants for solid lubrication applications, we conducted unidirectional sliding friction experiments with bonded molybdenum disulfide (MoS_2) films, magnetron-sputtered MoS_2 films, ion-plated silver films, ion-plated lead films, magnetron-sputtered diamondlike carbon (DLC) films, and plasma-assisted, chemical-vapor-deposited DLC films in contact with AISI 440C stainless steel balls in ultrahigh vacuum, in humid air, and in dry nitrogen. When the main criteria for judging the performance of the dry solid lubricating films were coefficient of friction and wear rate, which had to be less than 0.3 and 10^{-6} mm^3/N·m, respectively, the following remarks can be made:

1. Bonded MoS_2 and magnetron-sputtered MoS_2 films met both criteria in all three environments. Also, the wear rates of the counterpart 440C stainless steel balls met that criterion in the three environments.
2. In ultrahigh vacuum the coefficient of friction and endurance (wear) life of bonded MoS_2 films were superior to those of all the other dry solid film lubricants.
3. Ion-plated silver films met both criteria only in ultrahigh vacuum, failing in humid air and in dry nitrogen, where the film and ball wear rates were higher than the criterion.
4. Ion-plated lead films met both criteria only in ultrahigh vacuum, failing in humid air and in dry nitrogen, where the coefficients of friction were greater than the criterion. Both the lead film wear rate and the ball wear rate met that criterion in all three environments.
5. MS DLC and PACVD DLC films met the criteria in humid air and in dry nitrogen, failing in ultrahigh vacuum, where the coefficients of friction were greater than the criterion.
6. Adhesion and plastic deformation played important roles in the friction and wear of all the solid lubricating films in contact with 440C stainless steel balls in the three environments. All sliding involved adhesive transfer of materials: transfer of solid lubricant wear debris to the counterpart 440C stainless steel, and transfer of 440C stainless steel wear debris to the counterpart solid lubricant.

ACKNOWLEDGMENT

The author thanks M. Iwaki, K. Gotoh, S. Obara, and K. Imagawa of the Tsukuba Space Center, Tsukuba, Ibaraki, Japan, for furnishing the selected solid lubricating films; and M. Murakawa, S. Watanabe, and S. Takeuchi of the Nippon Institute of Technology for depositing the PACVD DLC films.

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TABLE I.—CHARACTERISTICS OF SELECTED SOLID LUBRICATING FILMS

[Substrate material, 440C stainless steel.]

Film	Film material	Film thickness, μm	Film surface roughness[a], R_a, nm	
			Mean	Standard deviation
Bonded MoS_2	MoS_2, polyimide-imide, others (proprietary blend)	10±4	1.2×10^3	2.4×10^2
MS MoS_2	MoS_2	1.0±0.2	32	4.0
Ion-plated silver	Silver	0.5±0.2	30	3.2
Ion-plated lead	Lead	0.55	98	15
MS DLC	Carbon and WC	2 to 3	43	5.1
PACVD DLC	Carbon and Si	3 to 5	29	3.2

[a] The centerline-average roughness R_a was measured by using a cutoff of 1 mm.

TABLE 2.—CONDITIONS OF BALL-ON-DISK SLIDING FRICTION EXPERIMENTS

Load, N	5.9
Sliding velocity, m/s	0.2
Vacuum pressure, Pa (torr)	10^{-7} (10^{-9})
Ball material and grade	440C stainless steel (grade 10)
Ball diameter, mm	6
Ball centerline-average surface roughness, R_a, nm	6.8 (with standard deviation of 1.8)
Ball average Vickers hardness number at loads from 0.49 to 4.9 N, GPa	8.7 (with standard deviation of 0.17)
Disk materials	Bonded MoS_2, MS MoS_2, ion-plated silver, ion-plated lead, MS DLC, PACVD DLC films on 440C stainless steel disk substrates

TABLE 3.—STEADY-STATE COEFFICIENT OF FRICTION, WEAR LIFE, AND WEAR RATE FOR SELECTED SOLID LUBRICATING FILMS IN SLIDING CONTACT WITH 440C STAINLESS STEEL BALLS

Film	Environment	Steady-state coefficient of friction	Film wear (endurance) life[a]	Film wear rate, $mm^3/N \cdot m$	Ball wear rate, $mm^3/N \cdot m$
Bonded MoS_2	Vacuum	0.045	>1 million	6.0×10^{-8}	1.3×10^{-9}
	Air	0.14	113 570	2.4×10^{-6}	8.1×10^{-8}
	Nitrogen	0.04	>1 million	4.4×10^{-8}	6.9×10^{-10}
MS MoS_2	Vacuum	0.070	274 130	9.0×10^{-8}	2.5×10^{-9}
	Air	0.10	277 377	2.4×10^{-7}	1.5×10^{-7}
	Nitrogen	0.015	>1 million	1.6×10^{-8}	9.9×10^{-10}
Ion-plated silver	Vacuum	0.20	364 793	8.8×10^{-8}	2.4×10^{-8}
	Air	0.43	8	5.5×10^{-5}	1.2×10^{-5}
	Nitrogen	0.23	1040	1.6×10^{-5}	1.6×10^{-5}
Ion-plated lead	Vacuum	0.15	30 294	1.5×10^{-6}	7.6×10^{-7}
	Air	0.39	82	3.7×10^{-6}	3.6×10^{-7}
	Nitrogen	0.48	1530	9.1×10^{-6}	3.4×10^{-6}
MS DLC	Vacuum	0.70	<10	5.7×10^{-5}	3.2×10^{-4}
	Air	0.12	$>3\times10^5$	1.7×10^{-7}	4.1×10^{-8}
	Nitrogen	0.12	23 965	4.2×10^{-7}	1.1×10^{-7}
PACVD DLC	Vacuum	0.54	<10	1.1×10^{-5}	1.8×10^{-4}
	Air	0.07	$>3\times10^5$	1.0×10^{-7}	2.3×10^{-8}
	Nitrogen	0.06	$>3\times10^5$	1.1×10^{-8}	6.4×10^{-9}

[a] Film wear life was determined to be the number of passes at which the coefficient of friction rose to 0.30.

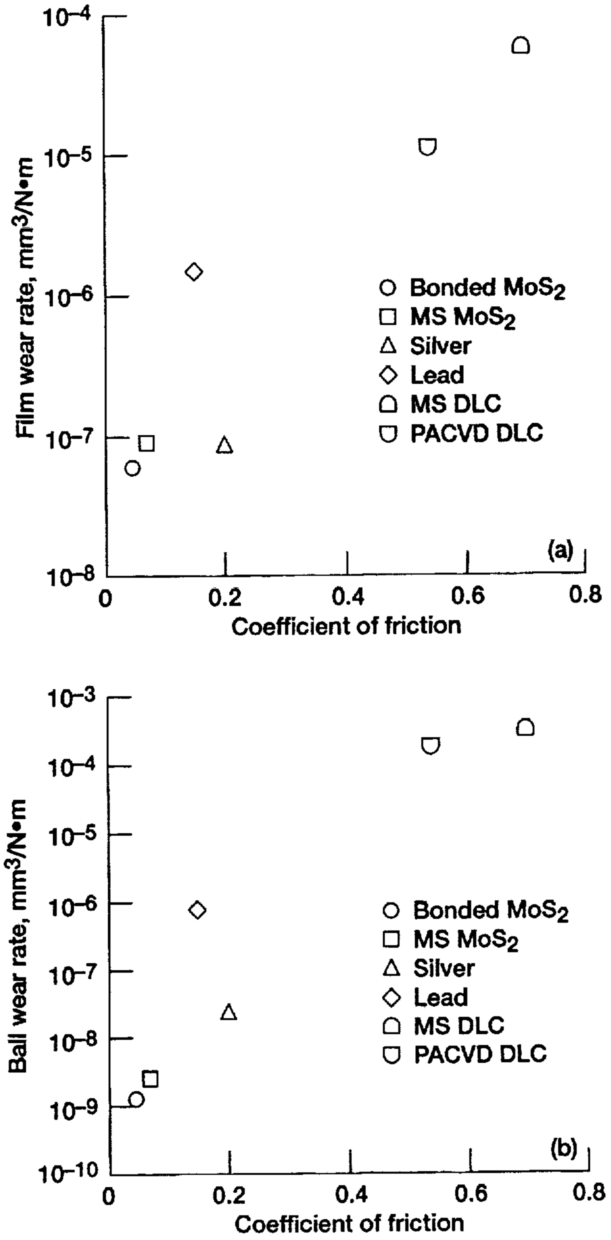

Figure 1.—Steady-state (equilibrium) coefficients of friction and wear rates (dimensional wear coefficients) for solid lubricating films in sliding contact with 440C stainless steel balls in ultrahigh vacuum. (a) Film wear rate. (b) Ball wear rate.

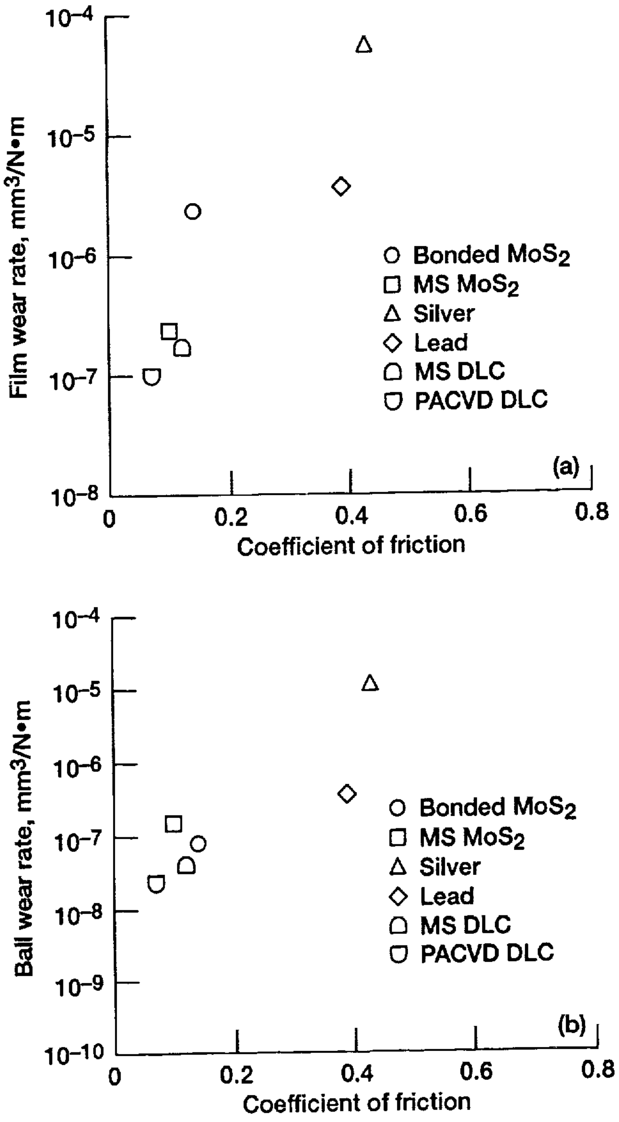

Figure 2.—Steady-state (equilibrium) coefficients of friction and wear rates (dimensional wear coefficients) for solid lubricating films in sliding contact with 440C stainless steel balls in humid air. (a) Film wear rate. (b) Ball wear rate.

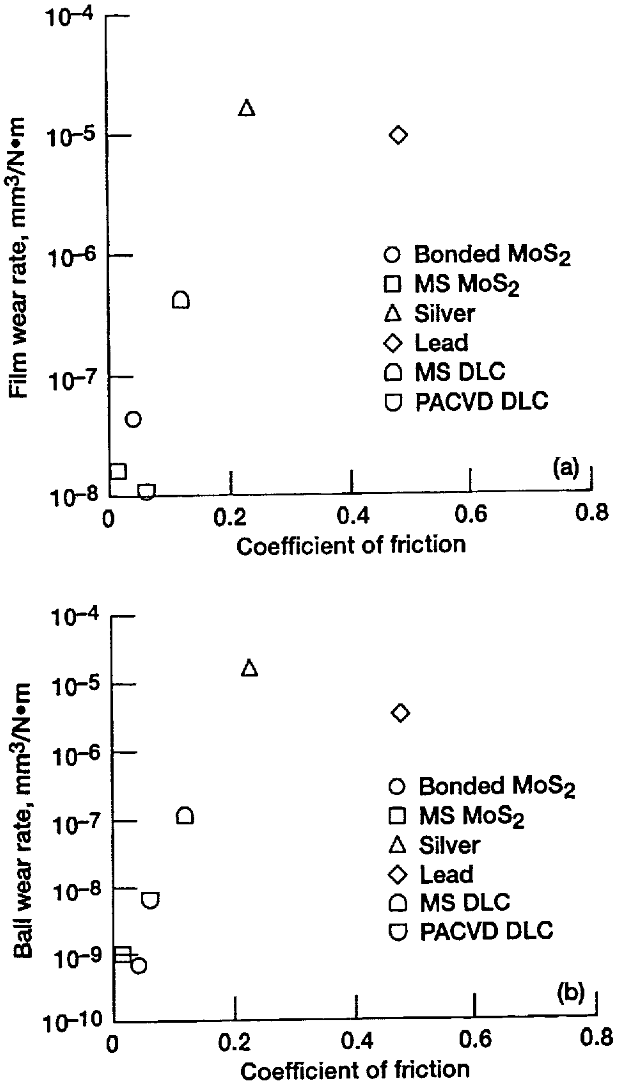

Figure 3.—Steady-state (equilibrium) coefficients of friction and wear rates (dimensional wear coefficients) for solid lubricating films in sliding contact with 440C stainless steel balls in dry nitrogen. (a) Film wear rate. (b) Ball wear rate.

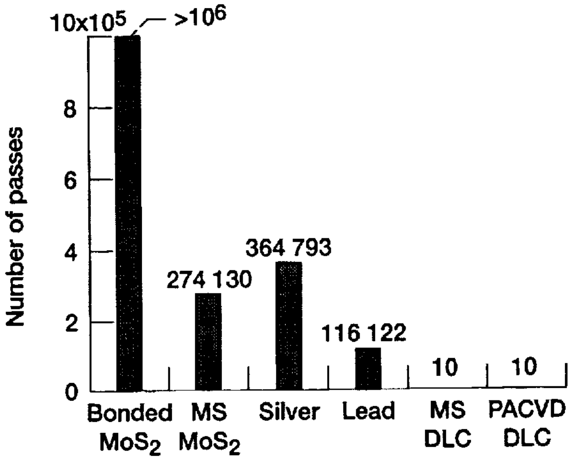

Figure 4.—Sliding wear lives for solid lubricating films in sliding contact with 440C stainless steel balls in ultrahigh vacuum.

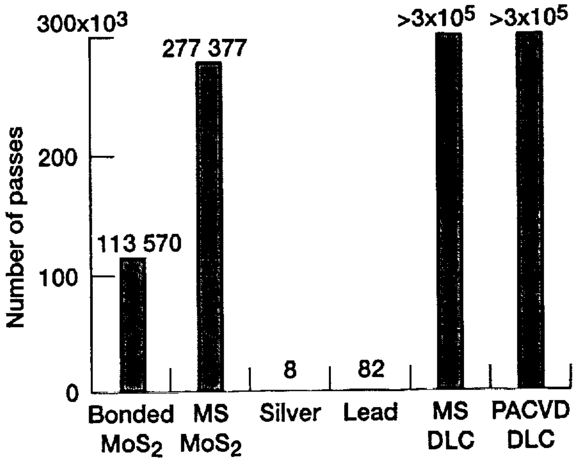

Figure 5.—Sliding wear lives for solid lubricating films in sliding contact with 440C stainless steel balls in humid air.

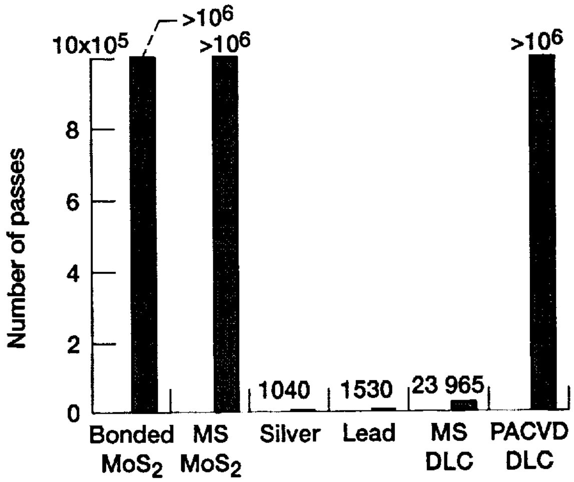

Figure 6.—Sliding wear lives for solid lubricating films in sliding contact with 440C stainless steel balls in dry nitrogen.