Durability Evaluation of Selected Solid Lubricating Films

Kazuhsa Miyoshi
Glenn Research Center, Cleveland, Ohio
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Kazuhisa Miyoshi
Glenn Research Center, Cleveland, Ohio

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DURABILITY EVALUATION OF SELECTED SOLID LUBRICATING FILMS

Kazuhisa Miyoshi
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

SUMMARY

An investigation was conducted to examine the coefficients of friction, wear rates, and durability of bonded molybdenum disulfide (MoS₂), magnetron-sputtered MoS₂, 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, $7 \times 10^{-7}$ 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 $10^{-6}$ mm³/N·m or less, respectively. The bonded MoS₂ and magnetron-sputtered MoS₂ 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 dry nitrogen. The MS DLC and PACVD DLC films met the requirements in humid air and dry nitrogen but failed in ultrahigh vacuum.

INTRODUCTION

Solid lubricants designed for spacecraft applications must not only display desirable coefficients of friction (0.001 to 0.3) but also maintain good durability in different environments, such as the atmosphere and high or ultrahigh vacuum. However, very little data are available on the tribological properties, particularly the actual wear rates, of solid lubricating films in different environments. The tribological properties of dry, solid lubricating films vary with the environment, possessing a Jekyll-and-Hyde character.

For many years it has been known that molybdenum disulfide (MoS₂), silver, lead, and carbon have outstanding lubricating properties. These materials are at the same time old lubricants and also the newest durable lubricants. The technology of solid lubrication has advanced rapidly in the past four decades, conforming primarily to the needs of the automobile and aerospace industries (refs. 1 to 12). Recently, a number of new applications, such as microelectromechanical systems (MEMS), gears and bearings for long-term service in space mechanisms, lightweight gear and bearing systems, and piston rings for low-heat-rejection engines, have arisen that have prompted renewed interest in solid lubrication. The new requirements are primarily long-term life and broad-temperature-range capability of solid lubricating coatings. However, the MEMS industry confronts significant adhesion, friction, and wear problems in manufacturing and actual use. New solid lubricants and their coating technologies are needed to meet these requirements and solve these problems.

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 lubricant. 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 objective of this study was to evaluate friction and wear properties, such as coefficient of friction, wear rate (dimensional wear coefficient), and endurance life, of selected solid lubricating films in three environments. Six types of newly developed, affordable, dry, solid film lubricant were evaluated: a bonded MoS₂, a magnetron-sputtered MoS₂, an ion-plated silver, an ion-plated lead, a magnetron-sputtered diamondlike carbon (MS DLC), and a plasma-assisted, chemical-vapor-deposited diamondlike carbon (PACVD DLC). Some earlier work on the films is reported in the references (refs. 13 to 15).

**EXPERIMENTAL MATERIALS—SELECTED SOLID LUBRICATING FILMS**

Three specimens of four film types, bonded MoS₂, magnetron-sputtered MoS₂, ion-plated silver, and ion-plated lead, were produced on American Iron and Steel Institute (AISI) 440C stainless steel disk substrates. Three specimens of two film types, MS DLC and PACVD DLC, were produced on silicon wafers attached to the 440C stainless steel disks. Characteristics of the six types of solid film lubricant are presented in table I.

**Molybdenum Disulfide Films**

Molybdenum disulfide is a versatile and useful solid lubricant (refs. 1 to 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 both bonded and magnetron-sputtered MoS₂ films were evaluated. As shown in table I, the bonded MoS₂ films were relatively rough, having centerline-average roughness \( R_a \) of 1.2 µm, and the magnetron-sputtered MoS₂ 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 hard 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 (ref. 5). In this study ion-plated silver and ion-plated lead films (refs. 13 and 14) were evaluated. These films were relatively smooth, having \( R_a \) of 30 and 98 nm, respectively, and were ~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.

Magnetron-sputtered (MS) DLC and plasma-assisted (PA) CVD DLC films were evaluated. These films were relatively smooth, having \( R_a \) of 43 and 29 nm, respectively, and were ~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 (\( \text{CH}_4 \)) gas. The multilayer film comprised 20- to 50-nm-thick, alternating layers of WC and carbon. The Vickers hardness number was ~1000. The PACVD DLC films were prepared by using radiofrequency plasma and consisted of two layers, an ~2-µm-thick DLC top layer on an ~2-µm-thick silicon-DLC underlayer. The DLC top layer was deposited by using \( \text{CH}_4 \) 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 \( \text{CH}_4 \) and tetramethylsilane (\( \text{C}_4\text{H}_{12}\text{Si} \)) gases. The ratio of the concentration of \( \text{CH}_4 \) and \( \text{C}_4\text{H}_{12}\text{Si} \) used was 90:18 (std cm³/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

The friction and wear properties of the selected solid lubricating films were examined in ultrahigh vacuum (7×10⁻⁷ Pa), in humid air at a relative humidity of ~20 percent, and in dry nitrogen at a relative humidity of <1 percent. Unidirectional ball-on-disk sliding friction experiments were conducted with 6-mm-diameter AISI 440C stainless steel balls in sliding contact with the solid lubricating films at room temperature in each environment. The disks were regular octahedrons 19 mm wide across the flats and 3 mm thick. As the disk rotated, the ball scribed a circular wear track on the flat disk surface. The assembly on which the disk specimen was mounted permits rotation at various track diameters, ranging from 5 to 16 mm in this investigation. The 440C balls (grade 10) used were smooth, having $R_a$ of 6.8 nm with a standard deviation of 1.8 nm. The Vickers microhardness at 0.49 to 4.9 N for the 440C balls was 8.7 GPa.

Three to five sliding friction experiments were conducted on each film type in each environment. All experiments were performed at a load of 5.9 N and a sliding velocity of 0.2 m/s. The steady-state coefficients of friction, wear rates (dimensional wear coefficients), and endurance lives for the solid lubricating films and the wear rates for the 440C stainless steel balls were obtained by averaging the data taken from a minimum of three experiments at various track diameters on three specimens of each film type in each environment. 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

Figures 1 to 3 summarize the average values of 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 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 MoS₂ films had the lowest coefficient of friction, lowest film wear rate, and lowest ball wear rate. The MS MoS₂ films also had low coefficient of friction, low film wear rate, and low ball wear rate, similar to those for the bonded MoS₂. The film and ball wear rates in ultrahigh vacuum were in ascending order: bonded MoS₂ < MS MoS₂ < 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 MoS₂, MS MoS₂, 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 MoS₂ < bonded MoS₂ < 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 MoS₂ films had the lowest coefficient of friction. The bonded MoS₂, MS DLC, 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 MoS₂ < bonded MoS₂ < MS DLC < ion-plated lead < ion-plated silver. The coefficients of friction and wear rates of the balls followed a similar ascending order.
Endurance Life

The endurance (sliding wear) 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 average values of sliding endurance lives, presented in figures 4 to 6, 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 endurance life, over 1 million passes. The endurance lives of the solid lubricating films in ultrahigh vacuum were in descending order: bonded MoS$_2$ > ion-plated lead > PACVD DLC > MS MoS$_2$.

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 endurance lives, over 1 million passes. The endurance 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 endurance lives, over 1 million passes. The endurance 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 (refs. 13 to 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 (ref. 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 (ref. 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 (ref. 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 (ref. 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 (ref. 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 (ref. 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, micropits, 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**

Recently developed, dry, solid film lubricants were evaluated for solid lubrication applications by conducting unidirectional sliding friction experiments with bonded molybdenum disulfide (MoS₂) films, magnetron-sputtered MoS₂ 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 ultra-
high 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 all 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.

REFERENCES

TABLE 1.—CHARACTERISTICS OF SELECTED SOLID LUBRICATING FILMS
[Substrate material, 440C stainless steel.]

<table>
<thead>
<tr>
<th>Film</th>
<th>Film material</th>
<th>Film thickness, µm</th>
<th>Film surface roughness*, Ra, nm</th>
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<tr>
<td></td>
<td></td>
<td>Mean</td>
<td>Standard deviation</td>
<td></td>
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<td>Bonded MoS₂</td>
<td>MoS₂, polyimide-imide, others (proprietary blend)</td>
<td>10 ± 4</td>
<td>1.2×10³</td>
<td>440C stainless steel</td>
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<tr>
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<td>1.0 ± 0.2</td>
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<td>40</td>
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<tr>
<td>Ion-plated silver</td>
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<tr>
<td>Ion-plated lead</td>
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<td>MS DLC</td>
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<td>43</td>
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<td>PACVD DLC</td>
<td>Carbon and Si</td>
<td>3 to 5</td>
<td>29</td>
<td>3.2</td>
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*aThe centerline-average roughness Ra was measured by using a cutoff of 1 mm.
Figure 1.—Average values of 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.—Average values of 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.—Average values of 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.—Average values of endurance lives for solid lubricating films in sliding contact with 440C stainless steel balls in ultrahigh vacuum.

Figure 5.—Average values of endurance lives for solid lubricating films in sliding contact with 440C stainless steel balls in humid air.

Figure 6.—Average values of endurance lives for solid lubricating films in sliding contact with 440C stainless steel balls in dry nitrogen.
Durability Evaluation of Selected Solid Lubricating Films

Kazuhisa Miyoshi

National Aeronautics and Space Administration
John H. Glenn Research Center at Lewis Field
Cleveland, Ohio 44135–3191

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