Friction and Wear Properties of Selected Solid Lubricating Films
Part 2: Ion-Plated Lead Films

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Acknowledgments

This research activity was basic research conducted under the Reimbursable Space Act Agreement between NASA Glenn Research Center and K Systems Corporation for Measurements and Analysis of Aerospace Tribology for Various Materials.

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SUMMARY

To evaluate commercially developed dry solid film lubricants for aerospace bearing applications, an investigation was conducted to examine the friction and wear behavior of ion-plated lead films in sliding contact with 6-mm-diameter American Iron and Steel Institute (AISI) 440C stainless steel balls. Unidirectional sliding friction experiments were conducted with a load of 5.9 N (600 g), a mean Hertzian contact pressure of 0.79 GPa (maximum Hertzian contact pressure of 1.2 GPa), and a sliding velocity of 0.2 m/s. The experiments were conducted at room temperature in three environments: ultrahigh vacuum (vacuum pressure, \(7 \times 10^{-7} \text{ Pa}\)), humid air (relative humidity, \(~20\%\)), and dry nitrogen (relative humidity, <1\%). The resultant films were characterized by scanning electron microscopy, energy-dispersive x-ray spectroscopy, and surface profilometry.

Marked differences in the friction and wear of the ion-plated lead films investigated herein resulted from the environmental conditions. The main criteria for judging the performance of the ion-plated lead films were coefficient of friction and wear rate, which had to be less than 0.3 and on the order of \(10^{-6} \text{ mm}^3/\text{N} \cdot \text{m}\) or less, respectively. The ion-plated lead films met both criteria only in ultrahigh vacuum but failed in humid air and in dry nitrogen, where the coefficient of friction was higher than the criterion. Both the lead film wear rate and the ball wear rate met that criterion in all three environments.

Adhesion and plastic deformation played important roles in the friction and wear of the ion-plated lead films in contact with 440C stainless steel balls in the three environments. All sliding involved adhesive transfer of materials: transfer of lead wear debris to the counterpart 440C stainless steel and transfer of 440C stainless steel wear debris to the counterpart lead.

INTRODUCTION

Soft metals have a number of properties that make them attractive as solid lubricants for special situations. For example, in addition to their low shear strengths and ability to be applied as continuous films over harder substrates, soft metals are good conductors of heat and electricity and are stable in vacuum or when exposed to nuclear radiation (ref. 1).

Soft metal films can be deposited as lubricating films on harder substrates by conventional electroplating or by physical vapor deposition methods, such as evaporation, 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. 2).

Thin-metal-film lubrication is most relevant at high temperatures or in applications where sliding is limited (e.g., rolling-element bearings). Lead (Pb) films have been very successful for long-term, rolling-element bearing lubrication in space mechanisms (ref. 3). One advantage of lead films over those of silver or indium is the unavoidable presence of lead oxide (PbO), a reputedly good solid lubricant, within the films (refs. 1 and 4). Ion-plated lead films are used in such mechanisms as solar array drives in European satellites (ref. 5). Lead
films have been used for many years to lubricate rolling-element bearings on rotating anode x-ray tubes, on satellite parts operating in space, and on other equipment exposed to high temperatures and nuclear radiation.

As the multidisciplinary science of lubrication, friction, and wear, tribology is application oriented, with a rich methodology. Once the initial shortcomings relating to lubrication in design and application had been dealt with, it became increasingly clear that materials science and technology ranked equal with design in reducing friction and wear of machinery and mechanical components (ref. 6), particularly in the field of space mechanisms. In modern technology, the coefficient of friction and the wear rate are regarded to be widely variable, depending on operational variables, lubricants, substrate properties, and surface films (ref. 7). Therefore, testing is central (ref. 8), and particularly standard performance testing of solid lubricant systems is important for the lubricant manufacturer during development of new lubricating materials, for quality control during lubricant manufacture, and for providing quantitative criteria for manufacturing specifications.

For mechanism design engineers (i.e., users), compiling manufacturers’ standard test results for a number of lubricant formulations can aid in selecting the best lubricant for an application. However, such data can at best only narrow the field to a specific class of lubricants. To decide on the optimum lubricant formulation for a specific application, more custom-design testing, element testing, component testing, and full-scale testing are required. However, after choosing the optimum lubricant, 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, so that 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.

In the part 1 of the investigation (ref. 8) three types of selected solid lubricating film were examined 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. The three types were bonded molybdenum disulfide (MoS2) films, magnetron-sputtered MoS2 films, and ion-plated silver films.

The present investigation was conducted to examine the friction and wear properties of ion-plated lead films in the same manner as the part 1 investigation. Unidirectional pin-on-disk sliding friction experiments were conducted with American Iron and Steel Institute (AISI) 440C stainless steel balls in sliding contact with the solid lubricating films at room temperature 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. 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 investigation. Surface profilometry was used to determine the surface morphology, roughness, and wear of the coatings.

SELECTED MATERIALS

Three specimens of each of the two thicknesses of ion-plated lead film produced on 440C stainless steel disk substrates were used in this investigation (table I). The 0.55-µm-thick, ion-plated lead films were relatively smooth, and their centerline-average roughness \( R_a \), measured using a cutoff of 1 mm, was 98 nm with a standard deviation of 15 nm. The 0.35-µm-thick, ion-plated lead films were also relatively smooth, and their \( R_a \), measured using a cutoff of 1 mm, was 40 nm with a standard deviation of 9.5 nm. The 6-mm-diameter 440C stainless steel balls (grade number, 10) used were smooth, having an \( R_a \) of 0.025 µm with a standard deviation of 0.02 µm or less.

EXPERIMENT

The pin-on-disk tribometer used in the investigation was mounted in a vacuum chamber (fig. 1). Unidirectional pin-on-disk sliding friction experiments were conducted in ultrahigh vacuum (7×10^{-7} Pa), in humid air (~20 percent relative humidity), and in dry nitrogen (<1 percent relative humidity) at room temperature. All experiments were conducted with 6-mm-diameter 440C stainless steel balls in sliding contact with the ion-plated lead films deposited on 440C stainless steel substrate disks. All experiments were conducted with a load of 5.9 N (600 g) at a sliding velocity of 0.2 m/s. The mean Hertzian contact pressure of the 440C stainless steel substrates in contact with the 440C stainless steel balls was ~0.79 GPa (maximum Hertzian contact pressure, 1.2 GPa). The pin-on-disk tribometer can measure friction in vacuum, in humid air, and in dry nitrogen during sliding. The friction force was continuously monitored during the friction experiments.
The sliding wear life for the coatings (film wear life or film endurance life) in this investigation was determined to be the number of passes at which the coefficient of friction rose to 0.3 in a given environment. Wear can be quantified by measuring the wear scars and wear tracks on the specimens after the wear experiments. Film wear volumes were obtained by averaging the cross-sectional areas, determined from stylus tracings, measured across the wear tracks at a minimum of four locations in each wear track. Then, the average cross-sectional area of the wear track was multiplied by the wear track length. The wear rate, known as the dimensional wear coefficient, is defined as the volume of material removed at a unit load and in a unit sliding distance expressed as cubic millimeters per newton-meter.

RESULTS AND DISCUSSION

Friction Behavior

Figures 2 to 4 present typical friction traces obtained in ultrahigh vacuum, in humid air, and in dry nitrogen for the ion-plated lead films in sliding contact with 440C stainless steel balls as a function of the number of passes. The friction traces obtained in vacuum fluctuated (fig. 2). The coefficient of friction for the 0.55-µm-thick, ion-plated lead film increased to 0.3 at 30 294 passes, and the coefficient of friction for the 0.35-µm-thick, ion-plated lead film increased to 0.3 at 21 825 passes. In humid air (fig. 3) the coefficients of friction for both film thicknesses rose to 0.3 in a small number of passes, approximately 100. In dry nitrogen (fig. 4) the coefficients of friction for both film thicknesses gradually increased to 0.3 at approximately 1000 passes. Comparing the data taken in different environments (figs. 2 to 4) shows that the coefficients of friction and the endurance lives of the ion-plated lead films were much better in ultrahigh vacuum than in either humid air or dry nitrogen.

Wear Behavior

Figures 5 to 7 present SEM photomicrographs of wear scars on 440C stainless steel balls and wear tracks on the ion-plated lead films deposited on 440C stainless-steel disks after sliding contact in ultrahigh vacuum, in humid air, and in dry nitrogen, respectively. The SEM observations were made either at the end of wear life, at which the coefficient of friction rose to 0.3, or at the number of passes at which the coefficient of friction rose to 0.5.

The wear tracks on the lead films revealed that the sliding action usually generated a smooth wear surface, fine wear-debris particles, and agglomerated, pasty wear debris, regardless of the environment. The wear scars on the 440C stainless steel balls were generally smooth, regardless of the environment. Thin, smeared wear patches and particles of the lead films generally covered the smooth wear scars. Smeared tongues of thin, layered, agglomerated wear debris were also present. Most of the loose and smeared wear debris accumulated outside the wear scars.

Wear Life (Endurance Life)

As in part 1 of this investigation 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 0.3 (ref. 8). The sliding wear lives of the ion-plated lead films examined in this investigation (fig. 8 and table II) varied with the environment, being much longer in ultrahigh vacuum than in dry nitrogen and in humid air.

Comparison of Steady-State Coefficients of Friction and Wear Rates

Table II and figure 9 present the steady-state coefficients of friction, the wear rates for the ion-plated lead films, and the wear rates for the 440C stainless steel balls after sliding contact in all three environments. The data presented in the table reveal the marked difference in coefficient of friction resulting from the environmental conditions. Both the 0.55- and 0.35-µm-thick, ion-plated lead films had low coefficients of friction in ultrahigh vacuum but relatively high coefficients of friction in humid air and in dry nitrogen. The coefficients of friction investigated herein were, in ascending order, those in ultrahigh vacuum, in humid air, and in dry nitrogen.
The wear rates for the ion-plated lead films and for the counterpart 440C stainless steel balls were not susceptible to environment. The wear rates in ultrahigh vacuum were similar to those in humid air and in dry nitrogen.

**Sliding Wear, Wear Debris, and Transferred Wear Fragments**

Adhesion and plastic deformation played important roles in the friction and sliding wear of the ion-plated lead films in contact with the 440C stainless steel balls in all three environments. The worn surfaces of both the films and the balls contained wear debris particles. Examining the morphology and compositions of the worn surfaces by SEM and EDX provided detailed information about plastic deformation of the lead films, wear debris, and transferred wear fragments produced during sliding (figs. 10 to 15). Marked plastic deformation occurred in the lead films. Smeared, agglomerated wear debris accumulated around the contact border, particularly on the ends of wear scars. All sliding involved adhesive transfer of materials.

**Ultra-high-vacuum environment.**—Figure 10(a) presents a typical wear track on a 0.55-μm-thick, ion-plated lead film over which a 440C stainless steel ball has passed in ultrahigh vacuum leaving a small amount of transferred steel wear fragments. The fine asperities of the ion-plated lead film were flattened and elongated in the direction of sliding by plastic deformation, revealing a burnished appearance. Figure 11(a) presents a typical wear track on a 0.35-μm-thick, ion-plated lead film over which a 440C stainless steel ball has passed in ultrahigh vacuum. The wear track on the 0.35-μm-thick film was similar to that on the 0.55-μm-thick film.

The entire wear scar on the counterpart 440C stainless steel ball (figs. 10(b) and 11(b)) contained thick transferred layers (or sheets) of lead. Plate-like lead debris particles were found at the edges of the film wear track. Severe plastic deformation and shearing occurred in the lead film during sliding.

**Humid-air environment.**—Figure 12(a) presents a typical wear track on a 0.55-μm-thick, ion-plated lead film over which a 440C stainless steel ball has passed in humid air leaving a small amount of transferred steel wear fragments. The fine asperities of the ion-plated lead film were flattened and elongated in the direction of sliding by plastic deformation, revealing a burnished appearance. Figure 13(a) presents a typical wear track on a 0.35-μm-thick, ion-plated lead film over which a 440C stainless steel ball has passed in humid air. The wear track on the 0.35-μm-thick film was similar to that on the 0.55-μm-thick film.

The wear scar on the counterpart 440C stainless steel ball (figs. 12(b) and 13(b)) contained an extremely small amount of transferred lead debris particles. This result suggests that oxidation of lead during sliding in humid air may prevent large lead transfer. In addition to the small wear debris particles, plate-like lead debris was found at the edges of the film wear track. Severe plastic deformation and shearing occurred in the lead film during sliding.

**Dry-nitrogen environment.**—Figure 14(a) presents a typical wear track on a 0.55-μm-thick, ion-plated lead film over which a 440C stainless steel ball has passed in dry nitrogen leaving transferred steel wear fragments. The fine asperities of the ion-plated lead film were flattened and elongated in the direction of sliding by plastic deformation, revealing a burnished appearance. Figure 15(a) presents a typical wear track on a 0.35-μm-thick, ion-plated lead film over which a 440C stainless steel ball has passed in dry nitrogen. The wear track on the 0.35-μm-thick film was similar to that on the 0.55-μm-thick film.

The wear scar on the counterpart 440C stainless steel ball (figs. 14(b) and 15(b)) contained transferred lead plates and particles. In addition to the small wear debris, plate-like lead debris was found at the edges of the film wear track. Severe plastic deformation and shearing occurred in the lead film during sliding.

**CONCLUSIONS**

To evaluate commercially developed solid film lubricants for aerospace bearing applications, unidirectional sliding friction experiments were conducted with ion-plated lead films in contact with AISI 440C stainless steel balls in ultrahigh vacuum, in humid air, and in dry nitrogen. The main criteria for judging the performance of the lead 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·m or less, respectively. The following conclusions were drawn:

1. 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 of 0.3. Both the lead film wear rate and the ball wear rate met that criterion in all three environments.
2. Adhesion and plastic deformation played important roles in the sliding friction and wear of the ion-plated lead films in contact with 440C stainless steel balls in all three environments. All sliding involved adhesive transfer of materials: transfer of lead wear debris to the counterpart 440C stainless steel and transfer of 440C stainless steel wear debris to the counterpart lead film.

REFERENCES

TABLE II.—STEADY-STATE COEFFICIENT OF FRICTION, WEAR LIFE, AND WEAR RATE FOR ION-PLATED LEAD FILMS IN SLIDING CONTACT WITH 440C STAINLESS STEEL BALLS

<table>
<thead>
<tr>
<th>Film thickness, µm</th>
<th>Environment</th>
<th>Steady-state coefficient of friction</th>
<th>Film wear (endurance) lifea</th>
<th>Film wear rate, mm³/N/m</th>
<th>Ball wear rate, mm³/N/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.55</td>
<td>Vacuum</td>
<td>0.15</td>
<td>30 294</td>
<td>1.5×10⁻⁶</td>
<td>7.6×10⁻⁷</td>
</tr>
<tr>
<td></td>
<td>Air</td>
<td>0.39</td>
<td>82</td>
<td>3.7×10⁻⁶</td>
<td>3.6×10⁻⁷</td>
</tr>
<tr>
<td></td>
<td>Nitrogen</td>
<td>0.48</td>
<td>1530</td>
<td>9.1×10⁻⁶</td>
<td>3.4×10⁻⁶</td>
</tr>
<tr>
<td>0.35</td>
<td>Vacuum</td>
<td>0.23</td>
<td>21 825</td>
<td>1.0×10⁻⁶</td>
<td>8.4×10⁻⁶</td>
</tr>
<tr>
<td></td>
<td>Air</td>
<td>0.38</td>
<td>115</td>
<td>1.7×10⁻⁶</td>
<td>4.0×10⁻⁶</td>
</tr>
<tr>
<td></td>
<td>Nitrogen</td>
<td>0.42</td>
<td>693</td>
<td>1.2×10⁻⁶</td>
<td>6.1×10⁻⁶</td>
</tr>
</tbody>
</table>

Film wear life is determined to be the number of passes at which the coefficient of friction rose to 0.3.

Figure 1.—Pin-on-disk tribometer in vacuum chamber and conditions of unidirectional, pin-on-disk, sliding friction experiments.

<table>
<thead>
<tr>
<th>Load, N (g)</th>
<th>5.9 (600)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Hertzian contact pressure, GPa</td>
<td>0.79</td>
</tr>
<tr>
<td>Maximum Hertzian contact pressure, GPa</td>
<td>1.2</td>
</tr>
<tr>
<td>Ball specimen</td>
<td>6-mm-diameter 440C stainless steel ball</td>
</tr>
<tr>
<td>Sliding velocity, m/s</td>
<td>0.2</td>
</tr>
<tr>
<td>Environments</td>
<td>Vacuum, 7×10⁻⁷ Pa</td>
</tr>
<tr>
<td></td>
<td>Humid air (relative humidity, ~20%)</td>
</tr>
<tr>
<td></td>
<td>Dry nitrogen (relative humidity, &lt;1%)</td>
</tr>
</tbody>
</table>
Figure 2.—Friction traces for (a) 0.55-μm-thick, ion-plated lead film and (b) 0.35-μm-thick, ion-plated lead film in sliding contact with 440C stainless steel balls in ultrahigh vacuum.
Figure 3.—Friction traces for (a) 0.55-μm-thick, ion-plated lead film and (b) 0.35-μm-thick, ion-plated lead film in sliding contact with 440C stainless steel balls in humid air.
Figure 4.—Friction traces for (a) 0.55-μm-thick, ion-plated lead film and (b) 0.35-μm-thick, ion-plated lead film in sliding contact with 440C stainless steel balls in dry nitrogen.
Figure 5.—Wear tracks produced on ion-plated lead films and wear scars produced on 440C stainless steel balls in ultrahigh vacuum. (a) Materials pair of ion-plated lead film (0.55 μm thick) and 440C ball at 116 112 passes. (b) Materials pair of ion-plated lead film (0.35 μm thick) and 440C ball at 21 825 passes.
Figure 6.—Wear tracks produced on ion-plated lead films and wear scars produced on 440C stainless steel balls in humid air. (a) Materials pair of 0.55-μm-thick, ion-plated lead film and 440C ball at 19 526 passes. (b) Materials pair of 0.35-μm-thick, ion-plated lead film and 440C ball at 22 003 passes.
Figure 7.—Wear tracks produced on ion-plated lead films and wear scars produced on 440C stainless steel balls in dry nitrogen. (a) Materials pair of 0.55-μm-thick, ion-plated lead film and 440C ball at 8916 passes. (b) Materials pair of 0.35-μm-thick, ion-plated lead film and 440C ball at 22,001 passes.
Figure 8.—Sliding wear life of ion-plated lead films in sliding contact with 440C stainless steel balls in ultrahigh vacuum, in humid air, and in dry nitrogen.
Figure 9.—Steady-state (equilibrium) coefficients of friction and wear rates (dimensional wear coefficients) for ion-plated lead films and 440C stainless steel balls (a) in ultrahigh vacuum, (b) in humid air, and (c) in dry nitrogen.
Figure 10.—Morphology and elemental composition (a) of wear track produced on 0.55-µm-thick, ion-plated lead film and (b) of wear scar produced on 440C stainless steel ball in ultrahigh vacuum.
Figure 11.—Morphology and elemental composition (a) of wear track produced on 0.35-μm-thick, ion-plated lead film and (b) of wear scar produced on 440C stainless steel ball in ultrahigh vacuum.
Figure 12.—Morphology and elemental composition (a) of wear track produced on 0.55-μm-thick, ion-plated lead film and (b) of wear scar produced on 440C stainless steel ball in humid air.
Figure 13.—Morphology and elemental composition (a) of wear track produced on 0.35-μm-thick, ion-plated lead film and (b) of wear scar produced on 440C stainless steel ball in humid air.
Figure 14.—Morphology and elemental composition (a) of wear track produced on 0.55-μm-thick, ion-plated lead film and (b) of wear scar produced on 440C stainless steel ball in dry nitrogen.
Figure 15.—Morphology and elemental composition (a) of wear track produced on 0.35-μm-thick, ion-plated lead film and (b) of wear scar produced on 440C stainless steel ball in dry nitrogen.
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