SOLID LUBRICANTS

A SURVEY

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SOLID LUBRICANTS
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By
M. E. Campbell

Prepared under contract NASw-1998
by Midwest Research Institute
Kansas City, Missouri
Foreword

The Technology Utilization Office of the National Aeronautics and Space Administration has established a program for the rapid dissemination of information on technological developments that may have potential utility outside the aerospace community.

This survey includes the most recent developments and trends in the field of solid lubrication. Major NASA research and NASA-sponsored research in solid-film lubricants are reviewed and various aspects of solid lubrication are discussed including: lubricating solids, bonded lubricants, new developments, methods of evaluation, environmental effects, application methods, novel materials, and designs for the use of solid lubricants. The appendices deal with solid-lubricant specifications and contact stresses imposed on specimens in three types of test machines. The information in this survey is of a practical nature, being derived from the experience of a number of investigators and users.

This survey, along with an extensive list of references and a bibliography should be useful to the many small companies that are not acquainted with the growth in scope and usage of solid lubricants. Data are summarized so that a broad spectrum of readers can more fully understand the mechanism of solid lubrication. Additionally, the publications cited in this document will enable the reader to obtain more specific information.

Director,
Technology Utilization Office
Acknowledgments

This document was prepared by a member of the staff of Midwest Research Institute. The author's knowledge in the field of solid lubrication was supplemented by information from solid-lubricant suppliers, NASA reports, personal contacts with NASA employees, and reports of and personal contacts with other U.S. Government agencies. The author particularly wishes to acknowledge the useful contributions of the engineers and scientists of the various NASA centers. The assistance of C. R. Brown of E. I. duPont de Nemours and Co., Inc. in preparing the section on "Lubricating Plastics" is also greatly appreciated.

Illustrations were obtained from E. I. duPont de Nemours and Co., Inc. (figs. 3 to 17); Faville-Levally Corp. (figs. 18 and 19); Dow Corning Corp. (figs. 22, 23, 36, 37, 45, and 46); and Hohman Plating and Manufacturing, Inc. (figs. 24 and 25).
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<td></td>
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<td>78</td>
</tr>
</tbody>
</table>
Introduction

The technology of solid lubrication has advanced rapidly in the past decade due primarily to the needs of the aerospace industry. Many applications are very specialized and oriented toward conditions imposed by high altitude and space environment. The significant growth in solid-lubricant technology has led NASA to look seriously at the potential for nonaerospace applications. Because much solid-lubricant data are generated by the government and, in some cases, under proprietary restrictions, they are not always readily available to the commercial lubrication engineer. In this survey, solid-lubricant information has been collected from all available sources, and is presented in a summary form that will help a broad spectrum of readers to better understand the mechanism of lubrication by solid materials.

The survey is divided into several chapters covering various aspects of solid lubrication. Topics discussed include: a history of solid lubrication, lubricating solids, bonded lubricants, new developments, methods of evaluation, environmental effects, application methods, novel materials, and designs for the use of solid lubricants. Excerpts of solid lubricant specifications and a discussion of contact stresses imposed on specimens in three types of test machines used for the evaluation of solid lubricants are found in the appendices.

A profuse amount of literature is available on solid lubrication. Not all of the literature is easily accessible to the layman, however, particularly that which has been generated by recent government sponsored research and development programs. The information contained herein was gathered from solid-lubricant suppliers, NASA reports, personal contacts with NASA employees at various NASA centers, reports of and personal contacts with other U.S. Government agencies, and from the author's knowledge and experience in the field of solid lubrication.

HISTORY OF SOLID LUBRICATION

The use of lubricants probably dates back to the invention of the wheel. Since this early beginning, lubrication and the production of lubricating media have grown to be one of the largest industries in
the world, yet from one-third to one-half of all the energy produced still is lost through friction. Until the early 1800's, animal fats and oils were the primary lubricant materials. Since the first oil well was drilled in Titusville, Pa., in 1859, the technology of lubrication has advanced rapidly. During the mid-1930's petroleum oils were improved through the use of additives, which increased the load-carrying ability, lubricity, corrosive protection, and thermal oxidative stability of oils.

A trend also developed that required moving parts to operate at higher and higher temperatures. Because petroleum oils could not adequately do the job at these high temperatures, synthetic lubricant materials were introduced. Temperatures now encountered in supersonic aircraft, spacecraft, and certain industrial applications are beyond the useful range of even the synthetic lubricants. This trend to the operation of bearing surfaces at higher temperatures and low pressures has led to the development and use of solid lubricants to attain the necessary lubrication. Solid lubricants have at least one very desirable feature—they do not evaporate under the aforementioned conditions. Although the slippery feel and appearance of graphite and molybdenum disulfide have been known for centuries, their use as solid lubricants dates back only about 25 years. Graphite is also known as black lead and plumbago. It was long confused with similar appearing minerals, particularly molybdenite (MoS₂), and was not classified as a separate mineral until 1556. In 1779 it was proved to be carbon when it was oxidized to carbon dioxide.

About 1564, the Borrowdale graphite mines in England began producing graphite for pencils. These early pencils were made by encasing slabs of cut graphite in slotted wooden dowels. The name graphite did not come into being until 1789, when Werner drew it from the Greek word graphein, which means to write.

The ore molybdenite (MoS₂) was known to the early Greeks. It has often been confused with graphite and with lead. The name is derived from the Greek word meaning lead.

Molybdenum is widely distributed over the earth's crust in the form of molybdenite. The largest commercial source of the mineral is in Climax, Colo., where it is mined from granite containing the ore in a finely divided state. Molybdenum disulfide has a metallic luster and is blue-grey to black in color. Early pioneers traveling through the Climax area used pulverized rock to lubricate the wheels on their Conestoga wagons. This probably was one of the first uses of a molybdenum disulfide as a solid lubricant in this country.

A solid lubricant can generally be defined as a material that provides lubrication to two relatively moving surfaces under essentially
dry conditions. The most common of the present dry solid lubricants are graphite and molybdenum disulfide. Other materials that have been considered for use as solid lubricants, as well as coefficient of friction data for those materials, are listed in tables I, II, and III. The friction values given in the tables are higher than the values often quoted in the literature for solid lubricant films. This difference in values can be attributed to the fact that the reported values were determined under light-load conditions (50 psi or less), whereas values quoted for thin films are usually at higher loads (1000 psi or more). An example of this can be seen in comparing the value of 0.34 reported for MoS₂ with the values of 0.12 to 0.04 obtained on a thin MoS₂ film as load is increased from 1000 to 150,000 psi. For convenient testing, the materials listed in tables I, II, and III were compacted into pellets; tests were then conducted to determine the relative lubricative merits of the materials. Pelletizing of candidate lubricant materials has been widely used for obtaining comparative friction information.

Although the use of graphite as a lubricant probably began in the middle ages, the use of bonded solid-lubricant materials discussed in this document is relatively new. The first U.S. patent for bonded materials (phosphoric acid bonded graphite film) was issued in the middle 1940's. (Patents covering bonded solid-film lubricants usually are issued covering both the lubricating solid and the binder material—the medium by which the lubricating solid is attached to the substrate.) Several hundred patents for solid-lubricating materials and binders have been issued so far.

The use of molybdenum disulfide as a lubricating solid also started in the 1940's and it is now used in more applications than any of the other lubricating solids. A large variety of binder materials was evaluated in the early bonded-film work, including materials such as corn syrup, asphalt-base varnish, silicone-base varnish, and glycerol (ref. 1). Binder materials now include thermoplastic and thermosetting resins, metals, ceramics, and metal salts. Soft metals, metallic oxides, metallic sulfides, metallic selenides, metallic tellurides, and many others are currently being considered as solid lubricants by a large number of research groups.

The study of solid lubricants as now known is a relatively new field. No systematic study of these materials was begun until long after they were introduced in the aircraft industry. Solid lubricants sometimes were sold as “cure-all” materials, resulting in many misapplications. Unfortunately, the misapplications outweighed the proper applications, thereby slowing down the general acceptance of these lubricants by industry. Designers still should not consider the use of solid lubricants in many areas.
### Table I.—Friction Data for Compounds Considered as Solid Lubricants

<table>
<thead>
<tr>
<th>Material</th>
<th>Load g</th>
<th>Coefficient of Friction</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>80°F</td>
<td>500°F</td>
</tr>
<tr>
<td>Ag₂Se</td>
<td>326</td>
<td>0.30-0.35</td>
</tr>
<tr>
<td>LiF</td>
<td>&quot;</td>
<td>0.3-0.4</td>
</tr>
<tr>
<td>Si₃N₄</td>
<td>&quot;</td>
<td>0.3-0.9</td>
</tr>
<tr>
<td>SrS</td>
<td>&quot;</td>
<td>0.7-0.9</td>
</tr>
<tr>
<td>Tl₂S</td>
<td>&quot;</td>
<td>0.25-0.5</td>
</tr>
<tr>
<td>ZnSe</td>
<td>&quot;</td>
<td>0.4-0.6</td>
</tr>
<tr>
<td>ZrN</td>
<td>&quot;</td>
<td>0.2-0.3</td>
</tr>
<tr>
<td>AgI</td>
<td>128</td>
<td>1.0</td>
</tr>
<tr>
<td>Ag₂S</td>
<td>&quot;</td>
<td>0.36</td>
</tr>
<tr>
<td>Ag₂Te</td>
<td>&quot;</td>
<td>0.44</td>
</tr>
<tr>
<td>AlPO₄</td>
<td>&quot;</td>
<td>1.33</td>
</tr>
<tr>
<td>AlPO₄</td>
<td>&quot;</td>
<td>1.0</td>
</tr>
<tr>
<td>AlPO₄</td>
<td>326</td>
<td>0.60</td>
</tr>
<tr>
<td>Bi₂S₅</td>
<td>128</td>
<td>0.23-0.60</td>
</tr>
<tr>
<td>Bi₂S₅</td>
<td>326</td>
<td>—</td>
</tr>
<tr>
<td>Bi₂S₅</td>
<td>&quot;</td>
<td>0.56-0.58</td>
</tr>
<tr>
<td>Bi₂S₃ + Bi₂O₃</td>
<td>128</td>
<td>0.38</td>
</tr>
<tr>
<td>Bi₂S₃ + Bi₂O₃</td>
<td>&quot;</td>
<td>—</td>
</tr>
<tr>
<td>CdSe</td>
<td>128</td>
<td>0.23-0.33</td>
</tr>
<tr>
<td>CdS</td>
<td>&quot;</td>
<td>0.58-1.0</td>
</tr>
<tr>
<td>CdTe</td>
<td>&quot;</td>
<td>0.46</td>
</tr>
<tr>
<td>CoS</td>
<td>&quot;</td>
<td>0.50-1.15</td>
</tr>
<tr>
<td>Cu₂S</td>
<td>128</td>
<td>1.0</td>
</tr>
<tr>
<td>GeO₂</td>
<td>&quot;</td>
<td>0.48-0.58</td>
</tr>
<tr>
<td>InSe</td>
<td>&quot;</td>
<td>0.46-0.60</td>
</tr>
<tr>
<td>MoSe₂</td>
<td>&quot;</td>
<td>0.20-0.33</td>
</tr>
<tr>
<td>NiS</td>
<td>&quot;</td>
<td>0.29-1.0</td>
</tr>
<tr>
<td>PbSe</td>
<td>&quot;</td>
<td>0.40-0.67</td>
</tr>
<tr>
<td>PbS</td>
<td>&quot;</td>
<td>0.08</td>
</tr>
<tr>
<td>PbS</td>
<td>&quot;</td>
<td>0.30-0.68</td>
</tr>
<tr>
<td>PbS(II)</td>
<td>326</td>
<td>0.27-0.39</td>
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<td>PbS</td>
<td>&quot;</td>
<td>0.47</td>
</tr>
<tr>
<td>PbS-MoS₂</td>
<td>&quot;</td>
<td>0.16-0.38</td>
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<td>PbS-graphite</td>
<td>&quot;</td>
<td>0.20</td>
</tr>
<tr>
<td>Sb₂O₃</td>
<td>128</td>
<td>0.21-0.96</td>
</tr>
<tr>
<td>Sb₂S₅</td>
<td>&quot;</td>
<td>0.38</td>
</tr>
<tr>
<td>Sb₂S₇</td>
<td>&quot;</td>
<td>0.31-0.80</td>
</tr>
<tr>
<td>Sb₂S₇</td>
<td>&quot;</td>
<td>0.50</td>
</tr>
<tr>
<td>TiTe₂</td>
<td>128</td>
<td>0.36-0.58</td>
</tr>
<tr>
<td>TiTe₂</td>
<td>&quot;</td>
<td>0.48-0.58</td>
</tr>
<tr>
<td>ZnTe</td>
<td>&quot;</td>
<td>0.60-0.68</td>
</tr>
<tr>
<td>ZrCl</td>
<td>&quot;</td>
<td>0.45</td>
</tr>
<tr>
<td>Graphite no. 2</td>
<td>128</td>
<td>0.15</td>
</tr>
<tr>
<td>Graphite no. 1</td>
<td>&quot;</td>
<td>0.15</td>
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### Table I—(Concluded)

<table>
<thead>
<tr>
<th>Material</th>
<th>Load g</th>
<th>Coefficient of Friction</th>
<th>80°F</th>
<th>500°F</th>
<th>1000°F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Graphite</td>
<td>326</td>
<td></td>
<td>0.14-0.30</td>
<td>0.06-0.12</td>
<td>0.20-0.27</td>
</tr>
<tr>
<td>MoS₂ + graphite (7% by weight)</td>
<td>128</td>
<td></td>
<td>0.20-0.25</td>
<td>0.11</td>
<td>0.22-0.56</td>
</tr>
<tr>
<td>PbS(II)</td>
<td></td>
<td></td>
<td>0.55-0.70</td>
<td>0.3-0.7</td>
<td>—</td>
</tr>
<tr>
<td>MoS₂ + graphite (31% C by weight)</td>
<td>128</td>
<td></td>
<td>0.16-0.21</td>
<td>0.12-0.14</td>
<td>0.13-0.16</td>
</tr>
<tr>
<td>MoS₂ + graphite (73% C by weight)</td>
<td>“</td>
<td></td>
<td>0.15-0.21</td>
<td>0.08-0.11</td>
<td>0.11-0.17</td>
</tr>
<tr>
<td>MoS₂</td>
<td>326</td>
<td></td>
<td>0.34</td>
<td>0.10</td>
<td>—</td>
</tr>
</tbody>
</table>

Test Conditions: Mark III Pellet Machine
Load: 326 g (avg approx 50 psi)
Speed: 600 ft/min
Track: polished, Rex AAA steel.
Tests were conducted at Midwest Research Institute (MRI).

### Advantages and Disadvantages of Solid Lubricants

Sliney (ref. 2) and other investigators (refs. 3 through 10) suggest a number of advantages and disadvantages of solid lubricants. Basically, the following are as summarized by Benzing (ref. 11).

#### Advantages

- Good stability at extreme temperatures and in chemically reactive environments
- Affords design advantages:
  1. Lighter weight, simplification, recirculating oil system with pump and heat exchangers is eliminated, and fewer seals are required
  2. Improved dynamic mechanical stability; bearings can be placed closer to heat sources allowing the use of shorter rotating shafts, thereby alleviating

#### Disadvantages

- Coefficient of friction generally higher than with hydrodynamic lubrication
- Some wear unavoidable because of solid sliding contact
- Film coatings have finite wear-lives
- Solid lubricants have no cooling capacity
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TABLE II.—Results of Lubricity Tests on Thermally Stable Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Room temperature</th>
<th>450°F</th>
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<tbody>
<tr>
<td>BN</td>
<td>0.3</td>
<td>0.15</td>
</tr>
<tr>
<td>CdCl₂</td>
<td>0.6</td>
<td>0.17</td>
</tr>
<tr>
<td>CrCl₃</td>
<td>0.2–0.3</td>
<td>—</td>
</tr>
<tr>
<td>PbF₂</td>
<td>0.6</td>
<td>0.6</td>
</tr>
<tr>
<td>MnCl₂</td>
<td>0.35</td>
<td>0.17</td>
</tr>
<tr>
<td>NiCl₂</td>
<td>0.45</td>
<td>0.19</td>
</tr>
<tr>
<td>SnS₂</td>
<td>0.9–0.45</td>
<td>—</td>
</tr>
<tr>
<td>SnO</td>
<td>0.95⁺</td>
<td>—</td>
</tr>
<tr>
<td>SnS</td>
<td>0.95⁺</td>
<td>0.63</td>
</tr>
<tr>
<td>Ta₂S₄</td>
<td>1.15</td>
<td>—</td>
</tr>
<tr>
<td>TiC</td>
<td>0.55</td>
<td>—</td>
</tr>
<tr>
<td>TiS₂</td>
<td>0.7</td>
<td>0.6</td>
</tr>
<tr>
<td>WS₂</td>
<td>0.7–1.6</td>
<td>0.2</td>
</tr>
</tbody>
</table>

Test Conditions: Mark III Pellet Machine
Load: 326 g (avg approx 50 psi)
Speed: 600 ft/min
Track: polished, Rex AAA.
Tests were conducted at MRI.

Advantages Disadvantages
problems of shaft critical speed
Can be used under high nuclear radiation conditions
Can be used under extremely high-load conditions

Solid lubricants are often used on components that are difficult to lubricate with conventional liquids and, in many cases, can provide lubrication for the life of the part. These lubricants can also be used to advantage in equipment where an accumulation of dust or dirt cannot be tolerated. Because certain solid lubricants are not impact sensitive in LOX, they have been used widely in this environment.

Although solid lubricants will probably never replace the home oil can, they will greatly extend the useful life of many devices the homeowner does not remember to oil anyway. Even on machines that are, and should continue to be, lubricated with conventional oils and greases, the proper application of solid lubricants can fur-
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TABLE III.—Friction Data for Mica-type Minerals

<table>
<thead>
<tr>
<th>Material</th>
<th>Chemical formula</th>
<th>Air</th>
<th>Vacuum, 1x10^-5 torr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pyrophyllite</td>
<td>[Al₆(OH)₂] Si₄O₁₀</td>
<td>0.51-0.89</td>
<td>0.10-0.27</td>
</tr>
<tr>
<td>Muscovite</td>
<td>[Al₆(OH)₂] K Al Si₃O₁₀</td>
<td>&gt;1.0</td>
<td>&gt;1.0</td>
</tr>
<tr>
<td>Talc</td>
<td>[Mg₃(OH)₂] Si₂O₁₀</td>
<td>0.13-0.89</td>
<td>0.21-0.72</td>
</tr>
<tr>
<td>Phlogopite</td>
<td>[Mg₃(OH)₂] K Al Si₃O₁₀</td>
<td>0.38-0.64</td>
<td>—</td>
</tr>
</tbody>
</table>

Note: Coefficients of friction determined on Mark IV pellet apparatus:
Load: 90 g
Speed: 41 cm/sec; temperature —80° F.
Tests were conducted at MRI.

ther extend service life by taking some of the finality out of the seemingly inevitable failure to maintain a regular effective lubrication maintenance program.

For the immediate future, these solid materials will not displace the more conventional lubricants so much as they will provide better lubrication for operating machinery. The proper application of solid lubricants will permit the successful operation of machinery under conditions of very high temperature, cryogenics, very high vacuum, nuclear radiation, extreme loads, and chemically reactive environments.

The selection of a solid lubricant for a specific application is a complex lubrication-engineering task. However, a discussion of the theory of lubrication with solid materials, such as molybdenum disulfide, graphite, lubricating plastics, and others, is beyond the scope of this document. The information stressed in this survey is practical, being derived from the experience of a number of investigators and users.

Because of a lack of proper terminology, and also for proprietary reasons, lubricants will be described by trade names in this survey. It has been impractical to include every product that is available, although no product was knowingly ignored. No effort was made to rate the lubricants according to any order of preference, and products named in a list or table will be presented in alphabetical order.
Bonded Solid Lubricants

Solid lubrication can be accomplished in several modes—lubricating powders, bonded films, and lubricant composites (metal- and plastic-based). This chapter is limited to a discussion of bonded solid lubricants, the most widely used mode. Bonded solid lubricants are distinguished from other solid lubricants in that they are attached to the substrate by means of a binder material—resin, silicate, or ceramic. A sketch showing surfaces coated with a bonded solid lubricant is shown in figure 1.

**RESIN-BONDED SOLID-FILM LUBRICANTS**

Resin-bonded films are currently the most widely used solid lubricants, including both “air-cured and “heat-cured” binder materials (air-cured and heat-cured refer to the method used in polymerizing the resin binder). The solid lubricant pigments used most frequently in resin-bonded films are: molybdenum disulfide (MoS₂), tungsten disulfide (WS₂), polytetrafluoroethylene (PTFE), and graphite.

The pigment may consist of one lubricating solid or a mixture of several, the function of which is to reduce the wear and lower the friction of the system being lubricated.

The binder holds the lubricating pigment to the metal surface so that the motion of parts does not bring about a complete loss of the pigment from the system. In the formulation of resin-bonded solid lubricants, the proper pigment-to-binder ratio is very important. The pigment-to-binder ratio, however, can vary widely with the particular resin used.

Certain factors can affect the overall performance of bonded films in any given situation. One is the condition of the metal surfaces being lubricated, which, in most cases, are generally changed or modified by some pretreatment to obtain the best film performance. Other factors involve variables directly related to the application of the film, such as spraying techniques. In addition to application factors, environmental conditions and the operating characteristics of the system being lubricated can seriously affect the film quality. These factors should be considered in the selection of a bonded solid lubricant for each application.
The resin-bonded solid lubricants are generally applied in thin films to the surfaces of the components being lubricated. In most cases, the surfaces have been pretreated in a manner that will depend on the substrate being used and the service for which the parts are intended. Resin-bonded solid-lubricant films can be applied by spray, dip, or brush; spray is usually the most satisfactory. The coating thickness should range from 0.0002 to 0.0008 in., the optimum being about 0.0005 in. A film that is too thick will be structurally weak and peel or flake off with a sliding motion under load; if, on the other hand, the film is too thin, premature failure may result due to rupture.

Several researchers (refs. 12 through 15) have determined optimum film thicknesses for resin-bonded films. Although some of the findings are contradictory, it appears that for high load a thinner film (0.0003 in. per surface) will give the longest wear-life. For lighter load conditions the thickness can be substantially increased. However, economics of the coating process (such as spray time and curing time) should enter into any decision involving the use of thicker films. A second area that must be considered if thick films are used is the generation of wear debris. Large amounts of wear debris are generated from thick films and some provision must be made for the removal of this debris from the bearing area. The removal of wear debris is discussed further in chapter 9.

The wear-behavior and wear-life characteristics of a resin-bonded solid lubricant are different from those of most other solid film lubricants. In its initial wear-in, it will exhibit relatively high wear which will become less with time. The initial high wear rate can be attributed to the loss of loose material from the surface of the film and the compaction of the film by the applied load. As running continues, the film will appear glossy or burnished. The best performance, lowest wear, and steadiest friction are obtained during this time.
Several investigations have been conducted on the effects of various conditions believed to affect the performance of a film. The hardness of the metal substrate has been one of the areas investigated. Crump (ref. 16) determined the beneficial effects of hardness on wear rate. Lavik (ref. 17) found little or no effect of hardness in the case where test specimens were of the line-contact type, whereas with the conforming-contact type he found a slight benefit with increased substrate hardness. Gaddis (ref. 18) found a substantial increase in wear-life with increased surface hardness on lightly loaded high-surface-speed tests. All of these programs were conducted on simulative bench testers in the laboratory. It was found that in hardware applications the harder bearings offer more beneficial results (longer film life with lower wear rates) than those obtained on softer substrates.

Bonded dry-film lubricants can provide long wear-life, good abrasion resistance, good adhesion, and good resistance to a variety of solvents. Performance of the films depends to a large extent on the cured properties of the binders used.

**Air-Cured Resin-Bonded Solid Lubricants**

An air-cured, resin-bonded solid lubricant consists of a lubricating powder, or powders, in an air-curing resin-binder material. The lubricating pigments most frequently used are molybdenum disulfide, graphite, or a lubricating plastic such as polytetrafluoroethylene. This type of solid film lubricant usually contains a lower total solid content than heat-cured film to provide a more satisfactory solution for aerosol application.

Binder materials used in the air-drying solid lubricants are thermoplastic resins, such as cellulosics and acrylics. These resins require no heat cure and therefore can be used on substrates that cannot be baked. They produce a fairly hard film, but do not have good solvent resistance. Examples of air-drying materials are:

Fel-Pro C-300 ........ Fel-Pro Inc., Skokie, Ill.


Molykote M88 ........ Dow Corning Corp., Stamford, Conn.

Molykote M8800 ....... Dow Corning Corp., Stamford, Conn.

Molykote 321 ........ Hohman Plating and Manufacturing, Inc.

Surf-Kote A-1625 ....... Hohman Plating and Manufacturing, Inc.

Dayton, O.
Heat-Cured Resin-Bonded Solid Lubricants

Heat-cured resin-bonded solid lubricants are the most widely used in the dry-film lubricant industry. The materials consist of the lubricating pigment and a specially formulated resin binder. The lubricating pigment is usually a mixture of approximately 90 percent molybdenum disulfide and 10 percent graphite, which seems to give the best results when friction and wear are considered. The relatively small concentration of graphite appears to improve the low-load performance (lower friction) of the MoS₂. Replacing graphite with antimony trioxide (Sb₂O₃) produces the same effect. Films are available that contain small percentages of such elements as silver, indium and lead, as well as a mixture of MoS₂ and graphite, but the lubricant coatings containing only graphite and molybdenum disulfide are more readily obtainable.

The frictional behavior of a material is an important consideration in its selection as a solid lubricant for resin-bonded films. Feng (ref. 19) indicates higher values of friction for a bonded film than for the lubricating solid itself. This may no longer be the case, since thermosetting resin-binder materials have been improved to the point where they do not appear to alter the friction values of the lubricating solids.

Curing of the binders in these films will usually require a bake of approximately 1 hr at 300° to 400° F. Special films such as those containing polyimide binders require baking temperatures of 575° F for 2 hr. Because of the baking temperature, care in the selection of the metal substrate is required. For example, temperatures of about 275° F for 1 hr can weaken certain aluminum alloys.

Binders that are normally used in the heat-cured solid lubricants are thermosetting and include alkyds, phenolics, epoxies, silicones, and polyimides. Alkyds are relatively inexpensive, cure at low temperatures (less than 500° F), and are generally easy to handle. Phenolics have good surface adhesion and are harder than the alkyds, but require a high-temperature curing cycle, usually 300° to 400° F for 1 hr. Epoxy resins have excellent solvent resistance and very good adhesion, but are softer than phenolics. Modified epoxy-phenolics combine the good properties of both materials. Silicones offer a higher operating temperature, but are softer and have only fair adhesion.

The polyimides are relatively new in the adhesive field. They were originally intended as laminating resins for use with fiber glass cloth. They have also been used as a wire insulation in electric motors where high temperature is a problem. The polyimide binder materials have extended the useful range of the resin-bonded lubri-
cant films up to approximately 700°F. Films containing these materials have been evaluated at temperatures up to 1000°F in vacuum (ref. 20). Such tests have demonstrated that the polyimide resins have a limited life at extreme temperatures. The polyimide-bonded solid-lubricant films are superior in extremely high-load applications. The polyimide-bonded films will be discussed further in chapter 3.

There are several other new binder materials being considered for use with solid lubricants. These materials are similar in structure to the polyimides and include the pyrrones, polybenzimidazoles (PBI) and polybenzothiazoles (PBT). Of these three materials, the pyrrones are very resistant to oxygen and strong acids.

These heat-cured materials are superior to the air-drying materials and should be used where high load-carrying ability or long life is required. They are usable over the temperature range of -100°F to +700°F. Examples of the heat-cured materials are:

- Drilube no. 1 . . . . . . . . . Drilube Co., Glendale, Calif.
- Molykote 106 . . . . . . . . . . Dow Corning Corp., Stamford, Conn.
- NPI 425* . . . . . . . . . . . . . National Products Industries, Southgate, Calif.
- Poxylube 500 M . . . . . . . . . Poxylube, Inc., Indianapolis, Ind.
- RIA no. 9A . . . . . . . . . . . . Sandstrom Products Co., Port Byron, Ill.
- Surf-Kote H 108 . . . . . . . . . Hohman Plating and Manufacturing, Inc., Dayton, O.

The importance of the resin-bonded solid lubricants has grown rapidly in the last 20 years. Because of this rapid growth, a means of controlling the quality of the bonded films was needed. To ensure that quality be maintained, government agencies have prepared specifications covering the materials and their uses. Typical of these specifications are MIL-L-8937, MIL-L-23398, and MIL-L-46010. There are also several custom variations available in these heat-cured, solid-film lubricants.

**INORGANIC-BONDED SOLID-FILM LUBRICANTS**

Inorganic-bonded lubricating pigments are usually referred to as high-temperature solid lubricants. These materials are intended for use at temperatures from approximately 500°F to in excess of

* Polyimide-bonded material MLR-2, developed by MRI under a NASA contract.
1200° F. There is considerable overlap in applicable temperature ranges for the various binder materials; however, certain ones operate very satisfactorily at temperatures down to -300° F. The high-temperature inorganic-bonded solid lubricants are a logical extension to the resin-bonded types. They employ ceramic or salt-based binders to give greater temperature resistance than resins and usually employ lubricating solids which are more thermally and oxidatively stable than graphite or MoS₂. These lubricants usually contain lubricating solids (pigments) such as lead oxide, lead sulfide, calcium fluoride, gold, and silver. There are exceptions, however, and a number of the ceramic and salt-based binders are used with MoS₂ and graphite.

Nonceramic Materials

The bonding technique for these materials commonly employs water-soluble silicates and phosphates, which produce a hard coating that tends to be somewhat brittle when cured (curing is accomplished by slowly driving out the excess water by heating). In general, they can be used at temperatures from -300° to +1000° F. Solid lubricants containing the salt-based binders usually contain graphite, MoS₂, lead sulfide, or powdered metals. They can be used in extremely high-load areas, with loads in excess of 100,000 psi. For applications where movement is a prime design consideration, however, they are not as good as resin-bonded films as far as wear-life and strength are concerned. Two advantages of these films over others are that (1) they will not outgas significantly in a vacuum of 10⁻⁹ torr and (2) they are compatible with liquid oxygen. Disadvantages include (1) lack of corrosion protection and (2) softening of the film in the presence of water or moisture for extended periods of time. Examples of salt-based bonded films are:

Drilube 805 . . . . . . Drilube Co., Glendale, Calif.
MLF-5* . . . . . Midwest Research Institute, Kansas City, Mo.
MLF-6* . . . . . Midwest Research Institute, Kansas City, Mo.
MLF-7* . . . . . Midwest Research Institute, Kansas City, Mo.
MLF-8* . . . . . Midwest Research Institute, Kansas City, Mo.
MLF-9* . . . . . Midwest Research Institute, Kansas City, Mo.

* Developed by MRI for the Marshall Space Flight Center on contract NAS8-1540.
Ceramic-Bonded Materials

The ceramic bonding agents are glasses rather than resins. Although glasses are liquid at all temperatures, they do not tend to flow perceptibly until heated above their softening point; this fact makes them prime candidates as binders for high-temperature solid lubricants. Their principal advantage is their good strength at elevated temperatures. The lubricating solids commonly employed with ceramic binders are graphite, calcium fluoride (CaF₂), lead oxide, and a mixture of barium fluoride (BaF₂) and CaF₂. Useful temperature ranges are from approximately 500°F to more than 1500°F.

Although the ceramic-bonded materials, as a class, do not perform as well (i.e., have low friction and wear), as the resin-bonded materials at lower (room) temperatures, they are generally better than the resin-bonded films by a considerable amount (over 10 times) at higher temperatures (700°F). There are exceptions, however, one being when the lubricants are run at high speeds, resulting in high temperatures (over 700°F) being generated in the contact zone. In such case, the ceramic-bonded films will generally outperform the resin-bonded films. The North American Rockwell Corp. has shown that their Vitrolube material outperforms the resin-bonded lubricants at room temperature (at low speed as well as high speed).

Examples of ceramic-bonded films are:

- NASA CaF₂ . . . . . . NASA-Lewis Research Center, Cleveland, O.

One problem in the use of ceramic-bonded materials is the thermal expansion of the cured coating. This must be matched closely with the expansion of the base material. If the thermal expansion characteristics are not the same or very similar, the coating will fracture and separate from the substrate.

PRETREATMENTS

It is standard practice to pretreat surfaces prior to the application of bonded solid lubricants. Pretreatments such as phosphating or grit blasting can greatly affect the performance of bonded solid lubricants by tending to increase the surface area and thus give the lubricant binder more area on which to hold. Also, these pretreat-

* Licensed and applied by National Process Industries, Southgate, Calif.
SOLID LUBRICANTS

ments generally increase the wear-life of the lubricant film. Whether chemical or mechanical pretreatments are used will depend on the base metal being employed. Pretreatments are required in nearly all of the solid-lubricant specifications described in appendix A.

Pretreatments for various metal surfaces to improve the wear-life of resin-bonded solid lubricants are given below.

<table>
<thead>
<tr>
<th>Type of metal</th>
<th>Pretreatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel</td>
<td>Grit blasting or other mechanical process</td>
</tr>
<tr>
<td></td>
<td>Phosphating</td>
</tr>
<tr>
<td></td>
<td>Sulfiding {</td>
</tr>
<tr>
<td></td>
<td>(except stainless steel)</td>
</tr>
<tr>
<td>Aluminum</td>
<td>Anodizing</td>
</tr>
<tr>
<td>Chromium and nickel plating</td>
<td>Vapor or grit blasting</td>
</tr>
<tr>
<td>Titanium</td>
<td>Vapor or grit blasting</td>
</tr>
<tr>
<td>Cadmium plate</td>
<td>Phosphating</td>
</tr>
<tr>
<td>Zinc plate</td>
<td>Phosphating</td>
</tr>
<tr>
<td>Copper and copper alloys</td>
<td>Bright dipping</td>
</tr>
<tr>
<td>Stainless steel</td>
<td>Chemical etching or grit blasting</td>
</tr>
</tbody>
</table>

There are other pretreatments that give acceptable behavior in specific applications, but the way in which these pretreatments improve the wear-life of a solid lubricant is not fully understood. The theory stated earlier, that pretreatment increases the surface area, is accepted by most workers in the field. Devine et al (ref. 21) suggest that pretreatment causes the formation of small pits or reservoirs of the lubricant which feed lubricant to the wearing area during the rubbing process. This theory is based on Devine's observations that reservoirs machined in a metal surface also give improved performance. Investigators at The Boeing Co. have done similar work with shot-peened surfaces which held large amounts of solid lubricant, and agree with Devine.

The most common pretreatments are phosphating, anodizing, and grit blasting. These have had widespread use, particularly in the aerospace industries where common bearing or wearing surfaces include steel, aluminum, and titanium. Phosphate coatings are used extensively with liquid lubricants to improve bearing life and to gain a small measure of protection from corrosion. They are also used under solid lubricants to gain some corrosion protection of the
substrate, since the solid lubricants do not provide such protection. Likewise, anodizing is preferred on aluminum parts for protection from corrosion.

Although proper phosphate pretreatments are difficult, Stupp (ref. 22) developed a good phosphate coating. He noted the effects of bath temperature, immersion time, and bath acid strength on the thickness of the coating, as well as the structure and size of crystal growth. Stupp found that the best conditions were: bath temperature—205° F, bath acid—7 to 8 points (full acid), and treatment time—approximately 15 min. The author is in complete agreement with these findings because he has often observed faulty solid lubricant coatings resulting from an improperly controlled phosphate bath. An important consideration in phosphate treatments is the temperature to which the part will be subjected in use. Gaddis (ref. 23) Campbell (ref. 24), and Lavik (ref. 25) have demonstrated that phosphate coatings should not be used at over 600° F.

The chemical pretreatments discussed above are used primarily in conjunction with the resin-bonded solid lubricants that have curing temperatures below 400° F. These same pretreatments can be used with the inorganic and nonceramic-bonded lubricants if the curing temperatures can be held below 400° F. The most accepted surface pretreatments for the inorganic-bonded films are wet- or dry-grit blasting. Pretreatment of surfaces for ceramic-bonded solid lubricants is in nearly all cases grit blasting. Sliney (ref. 26) has shown that a period of oxidation at 2000° F improves the bonding of ceramic-based solid lubricants on René 41 high-temperature alloy.

**APPLICATION PROCESSES**

The processes by which bonded solid lubricants are applied to bearing surfaces can have considerable effect on film behavior and performance. As a part of this survey, all modes of application—spraying, dipping, or brushing—of the various bonded films (resin, inorganic, nonceramic, and ceramic) will be discussed.

Of the three common methods of application, spraying and dipping are most often used. There are cases, however, where brushing can be used to advantage. The commonly held opinion that spraying a dry lubricant is just like spraying paint is not correct. The application of a solid lubricant in such a manner will result in a poor film. In a dipping process, the entire part is usually completely immersed in a lubricant bath, but this process will sometimes produce a film of nonuniform thickness. Generally speaking, a dry solid lubricant is a very special material and should be treated as such.

Table IV contains information on general processes for applying
TABLE IV.—Application Processes

Cleaning of components, including degreasing, and removal of dirt and corroded material

Steel (except stainless)
- Vapor, grit, or sandblast
- Phosphate

Aluminum and aluminum alloys
- Anodize or chemical film

Stainless steel
- Vapor, grit, or sandblast; chemical etch

Titanium
- Vapor, grit, or sandblast

Chrome or nickel plate
- Vapor, grit, or sandblast

Clean hot and cold water rinse
- Blow dry with clean dry air
- Oven dry
- Apply lubricant by brush, dip, or spray
- Oven cure

Notes:
(1) Parts to be dry lubricated should be handled only with clean, white cotton gloves after pretreatment operation.
(2) Continuous inspection is required during various processing steps.
(3) Dry-lubricated parts should be packaged separately as soon as possible after curing.
resin-bonded solid lubricants to various metal surfaces. Similar processes are outlined in Appendix A. Most manufacturers of bonded solid lubricants recommend certain methods of application for their particular films. Although these methods may be similar for various films, time and temperature for curing of the films will vary because of the different materials used in the preparation of the solid-lubricant formulations.

Accurate control of the many process variables is important. Control of such items as phosphating baths is essential, and the proper consistency of material to be sprayed must be maintained by the addition of proper thinners to the lubricant mixture prior to application. Curing temperatures also are important. In addition to controls at a given level, it is essential that the curing temperature not be so high as to affect the properties of the substrate material. Table V contains suggested maximum curing temperatures and times for resin-bonded solid lubricants on several materials. An undercured film will have poor adhesion and fluid resistance, while the coating cured too long at an extremely high temperature will probably have a reduced wear-life because the binder will suffer oxidation, thereby destroying the adhesion to the substrate material.

Films containing high-temperature polymeric binders of the polyimide type usually require much higher curing temperatures than those listed in table V. Curing temperatures for these materials can be as high as 575°F for 2 hr. Consequently, their use is limited to substrate materials that will not be weakened by the

<table>
<thead>
<tr>
<th>Substrate material</th>
<th>Temperature, °F</th>
<th>Time, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel and alloys</td>
<td>400</td>
<td>60</td>
</tr>
<tr>
<td>Steel and alloys, case hardened</td>
<td>300</td>
<td>60</td>
</tr>
<tr>
<td>Stainless steel (except 440)</td>
<td>400</td>
<td>60</td>
</tr>
<tr>
<td>440 stainless steel</td>
<td>300</td>
<td>60</td>
</tr>
<tr>
<td>Aluminum and magnesium alloys</td>
<td>275</td>
<td>60</td>
</tr>
<tr>
<td>Aluminum and magnesium alloys</td>
<td>300</td>
<td>25</td>
</tr>
<tr>
<td>Bronzes</td>
<td>300</td>
<td>60</td>
</tr>
<tr>
<td>Nickel and nickel alloys</td>
<td>400</td>
<td>60</td>
</tr>
<tr>
<td>Titanium</td>
<td>400</td>
<td>60</td>
</tr>
<tr>
<td>Monel and Inconel</td>
<td>400</td>
<td>60</td>
</tr>
<tr>
<td>Plated surfaces (chrome or nickel plated)</td>
<td>400</td>
<td>60</td>
</tr>
</tbody>
</table>
high-temperature cure. Curing of the inorganic-bonded films (such as silicate and phosphate) is accomplished in the temperature range of 300° to 400° F over extended periods of time, up to 16 hr. Certain of the ceramic-bonded films will require curing temperatures to 2000° F.

The equipment used in the application of bonded solid lubricants ranges from simple to very complex. The application process used will depend on the quantity and size of parts to be coated.
New Developments

The development of bonded solid lubricants has not always kept pace with the ever-changing requirements of industry. This has been particularly true in the aerospace industry where there is a need for lubricants that are usable over a wide variety of conditions. These conditions include: low and high temperatures, operation in vacuum for extended periods of time, and compatibility with oxidizers.

Many government agencies, including the Army, Navy, Air Force, Federal Aviation Administration (FAA) and NASA, are sponsoring research both in-house and under contract that is directed toward the development of new solid lubricants and techniques of lubrication. Most large aerospace companies also sponsor in-house programs on lubrication. This research has resulted in many significant contributions, and some new solid lubricants are now developed to the point where they are commercially available. Not all of the new developments can be covered in a publication of this nature, but this chapter will be devoted to a discussion of several of the newest materials.

**MLF- AND MLR-BONDED FILMS**

Midwest Research Institute has been conducting a program for NASA to develop, investigate, and evaluate solid-lubricant films suitable for use in both normal atmosphere and space environments. As a result of these programs, the bonded solid-lubricant films listed in table VI were developed.

The amount of development and testing already conducted on the MLF films (table VI) is so voluminous that it is impossible to report it in its entirety here. The complete history of these materials, however, can be found in references 27 through 32.

In the MLR series of solid lubricants, the MLR-1 and MLR-2 formulations use polyimide resin binders. These resins are stable at temperatures to 1000°F in air and over 1300°F in vacuum or in an inert atmosphere (ref. 20). The use of polyimide resin has increased the usable temperature range of resin-bonded solid lubricants from 450°F to over 700°F. In fact, when formulated with MoS₂ pigment
and used at elevated temperatures, it is possible to lose all of the solid lubricant pigment by oxidation and still have the binder material intact. The MLR–1 and MLR–2 lubricants are capable of carrying high loads for extended periods of time and also have low friction and long wear-life. Because the polyimide-resin binders require a high-temperature cure (575° F for 1 hr), they cannot be used on aluminum alloys or other metals that might be altered by the curing temperatures. The polyimide materials have been extensively investigated and are well documented in references 27 through 32.

The MLR–20 and MLR–21 materials listed in table VI are similar to the MLR–1 and MLR–2 materials in that they contain high-

Table VI.—Solid-Lubricant Film Compositions and Specifications

<table>
<thead>
<tr>
<th>Film designation</th>
<th>Lubricant-to-binder ratio</th>
<th>Ingredients and percent solids by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>MLF–5</td>
<td>10:1:5:7</td>
<td>MoS₂ (51), graphite (5), gold (26), sodium silicate (18)</td>
</tr>
<tr>
<td>MLF–6</td>
<td>10:1:3:7</td>
<td>MoS₂ (57), graphite (6), bismuth (17), sodium silicate (20)</td>
</tr>
<tr>
<td>MLF–7</td>
<td>10:1:5:5</td>
<td>MoS₂ (51), graphite (5), gold (26), potassium silicate (12), sodium fluoride (6)</td>
</tr>
<tr>
<td>MLF–8</td>
<td>10:1:5:8</td>
<td>MoS₂ (50), graphite (5), gold (25), aluminum phosphate (20)</td>
</tr>
<tr>
<td>MLF–9</td>
<td>10:1:14:9</td>
<td>MoS₂ (34), graphite (4), bismuth (47), aluminum phosphate (15)</td>
</tr>
<tr>
<td>MLR–1</td>
<td>1:0.54</td>
<td>MoS₂ (75), Sb₂O₃ (25), polyimide</td>
</tr>
<tr>
<td>MLR–2</td>
<td>1:0.54</td>
<td>MoS₂ (55), Sb₂O₃ (45), polyimide</td>
</tr>
<tr>
<td>MLR–20</td>
<td>1:0.27</td>
<td>MoS₂ (75), Sb₂O₃ (25), pyrrone</td>
</tr>
<tr>
<td>MLR–21</td>
<td>1:0.27</td>
<td>MoS₂ (90), graphite (10), pyrrone</td>
</tr>
<tr>
<td>MLR–30</td>
<td>10:0.21</td>
<td>MoS₂ (89), graphite (1), gold (5), glass resin</td>
</tr>
</tbody>
</table>

SPECIFICATIONS

MoS₂—Type Z and microsize, Dow Corning Corp., Stamford, Conn.
Graphite—No. 635, Joseph Dixon Crucible Co., Jersey City, N.J.
Gold—Fine powder 324 mesh, Goldsmith Brothers, Chicago, Ill.
Bismuth—Fine powder 325 mesh K 710, Electronic Space Products, Inc., Los Angeles, Calif.
Aluminum phosphate—50 percent solution, Stauffer Chemical Co., New York, N.Y.
Polyimide—PI4701 and PI1101, E. I. DuPont de Nemours and Co., Wilmington, Del.
Pyrrone—HR–100, Hughes Aircraft Co., Culver City, Calif.
Sb₂O₃ (reagent grade)—Fisher Scientific Co., Fair Lawn, N.J.
Glass resin—Type 908, Owens Illinois Glass Co., Toledo, O.
NEW DEVELOPMENTS

temperature polymeric binder materials. However, they do not exhibit all of the good qualities of the polyimide-bonded materials. At the same time, certain of their characteristics do offer some advantages. For example, it has been determined (ref. 24) that the pyrrone HR-100 material can be cured in a thin-film bonded solid lubricant at temperatures as low as 250°F. This lower cure temperature would allow their use in areas where the 575°F cure temperature used for the polyimide resins would be objectionable. Although the pyrrone-bonded materials are rather new, some information has been generated and is documented in reference 24.

Several organizations, including Midwest Research Institute, have investigated a series of glass-resin binders for use with solid-lubricant materials. One such material is the MLR-30 listed in table VI. The glass-resin-bonded solid lubricants do not exhibit extremely long wear-lives when evaluated on the standard Falex test machine, but they do offer several other advantages. These advantages are air curing and their ability to adhere to metal substrates without the necessity of elaborate pretreatment of the surface. Preliminary work on these materials has been conducted on a NASA contract (ref. 24).

CERAMIC-BONDED CALCIUM FLUORIDE SOLID LUBRICANT

As early as 1950, personnel at the Lewis Flight Propulsion Laboratory were experimenting with bonded solid-film lubricants. Much of their early work involved new methods of binding the lubricants to metal substrates and determination of film endurance characteristics (ref. 1). In the 1956-1958 period, they became involved in the development of lubricants for use in the 400°F to 1000°F temperature range. In one such investigation (ref. 33), lead oxide (PbO) was tested and found to be an effective lubricant in the range of 500°F to 1250°F. At 1250°F, this coating had a coefficient of friction that varied from 0.10 to 0.25, depending on the thickness of the applied film. The lowest friction and wear occurred with a coating 0.0005 in. thick. Experiments conducted at the Lewis Research Center indicated that this film had significantly longer endurance life at temperatures in excess of 500°F than at room temperature. Also, the room temperature endurance life was at least as good as that of a conventionally bonded MoS2 film tested under the same conditions. The testing of this film and of the CaF2 film described later was accomplished with equipment similar to that described in chapter 5 and shown in figure 2.

The PbO-SiO2 film did not become popular in the solid lubricant
industry, but it is probable that the material Electrofilm 1000 mentioned in chapter 2 is an outgrowth of this work.

In an effort to develop lubricants for use at still higher temperatures, the Lewis group initiated a program to investigate the feasibility of using various oxide and halide compounds as the lubricating solids in bonded solid-film lubricants (ref. 34). In these investigations the material had to be stable in air and of low volatility to 1500°F. Other properties considered important were high melting point, low water solubility, and crystal properties that are conducive to a low coefficient of friction, such as the presence of easily sheared lattice planes. The group concluded that of the materials investigated only CaF₂, BaF₂, and LiF merited further investigation.

Along with the investigation to determine the best solid lubricants, a program was conducted to develop binder materials. Coefficients of friction for the ceramic coatings were found to be between 0.3 and 0.4 over the range of 75°F to 1500°F. One ceramic composition containing a high percentage of cobalt oxide (CoO) was found to be an effective binder for the CaF₂ coating. The ceramic-bonded CaF₂ lubricant film (ref. 35) has now been patented (ref. 36). This film is recommended for use on nickel alloys over the temperature range of 500°F to 1900°F in both air atmosphere
and hard vacuum. The material can be thinned with water and applied by spraying, brushing, or dipping. A coating thickness of 0.0008 to 0.0035 in. is recommended, 0.001 to 0.002 in. being the most desirable.

As with the resin-bonded solid lubricants, pretreatment of the metal substrate will affect film performance. Oxidizing of the metal substrate in air at 2000° F, until a deep blue oxide film is formed, imparts the best endurance life to the film. An overlay of pure CaF₂ also increases wear-life but tends to increase the coefficient of friction. The composition of the films should be as shown in table VII (ref. 36).

<table>
<thead>
<tr>
<th>TABLE VII.—The Binder</th>
<th>Suitable range</th>
<th>Preferred range</th>
<th>Most preferred</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cobalt oxide (CoO)</td>
<td>40–70</td>
<td>55–65</td>
<td>60</td>
</tr>
<tr>
<td>Barium oxide (BaO)</td>
<td>10–30</td>
<td>15–25</td>
<td>20</td>
</tr>
<tr>
<td>Boric oxide (B₂O₃)</td>
<td>10–30</td>
<td>17–23</td>
<td>20</td>
</tr>
</tbody>
</table>

The pigment (lubricating solid CaF₂) to binder ratio can vary within wide limits, the most preferred ratio being 3:1. The effects of binder content on friction and wear at various temperatures are given in table VIII.

In further studies of the fluoride materials, Sliney (refs. 37 and 38) impregnated various nickel and nickel chromium alloys with a barium fluoride/calcium fluoride eutectic. The porous alloys were vacuum-impregnated with the fluoride composition at 2000° F. Friction and wear properties of the resultant bodies were determined in air and gaseous hydrogen at temperatures from 80° to 1500° F. Higher coefficients of friction were obtained on the composites than on dense metal substrates lubricated with coatings composed of the same fluoride composition. The advantages of coatings (low friction) and of the composites (long life) were combined by applying a thin sintered film of the eutectic fluoride to the porous composite. In a hydrogen atmosphere at a sliding velocity of 2000 ft/min, friction values ranged from 0.06 at 1500° F to 0.20 at 80° F. Friction values were higher in an air atmosphere, 0.20 over the range of 750° to 1500° F; at lower temperatures, the friction values increased to 0.35. Friction values also increased as sliding velocity decreased.
TABLE VII—Effects of Binder Content on Calcium Fluoride Solid Lubricant

Effect of ceramic content on coefficient of friction

<table>
<thead>
<tr>
<th>Binder Content</th>
<th>500°F</th>
<th>1000°F</th>
<th>1200°F</th>
<th>1500°F</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 part binder to 9 parts CaF₂</td>
<td>—</td>
<td>0.16</td>
<td>0.08</td>
<td>0.10</td>
</tr>
<tr>
<td>1 part binder to 4 parts CaF₂</td>
<td>0.30</td>
<td>0.18</td>
<td>0.07</td>
<td>0.08</td>
</tr>
<tr>
<td>1 part binder to 3 parts CaF₂</td>
<td>0.22</td>
<td>0.12</td>
<td>0.11</td>
<td>0.15</td>
</tr>
<tr>
<td>1 part binder to 1 part CaF₂</td>
<td>0.30</td>
<td>0.16</td>
<td>0.35</td>
<td>0.20</td>
</tr>
<tr>
<td>4 parts binder to 1 part CaF₂</td>
<td>0.49</td>
<td>—</td>
<td>—</td>
<td>0.15</td>
</tr>
</tbody>
</table>

Effect of ceramic content on endurance life (in number of test cycles to failure)

<table>
<thead>
<tr>
<th>Binder Content</th>
<th>500°F</th>
<th>1000°F</th>
<th>1200°F</th>
<th>1500°F</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 part binder to 9 parts CaF₂</td>
<td>—</td>
<td>14,000</td>
<td>38,000</td>
<td>17,000</td>
</tr>
<tr>
<td>1 part binder to 4 parts CaF₂</td>
<td>2,000</td>
<td>21,000</td>
<td>19,000</td>
<td>17,000</td>
</tr>
<tr>
<td>1 part binder to 3 parts CaF₂</td>
<td>(1)</td>
<td>(1)</td>
<td>(1)</td>
<td>29,000</td>
</tr>
<tr>
<td>1 part binder to 1 part CaF₂</td>
<td>24,000</td>
<td>(1)</td>
<td>25,000</td>
<td>28,000</td>
</tr>
<tr>
<td>4 parts binder to 1 part CaF₂</td>
<td>3,000</td>
<td>—</td>
<td>3,000</td>
<td>43,000</td>
</tr>
</tbody>
</table>

1 No failure after 50,000 cycles.

OTHER FLUORIDE FILMS

Researchers at Midwest Research Institute have developed a series of films designed to utilize the desirable friction and wear characteristics of the fluoride materials. They have found that the addition of monoaluminum phosphate to the lubricant enhances both its friction and wear-life characteristics.

The outstanding feature of these fluoride films is their ability to function effectively over a wide range of temperatures in both air and vacuum. Wear-lives of up to 1,000,000 load cycles have been obtained on these fluoride films when tested on the Mark V double rub-shoe tester described in chapter 5. The most effective temperature range for these materials has been found to be from 500°F to 1400°F. In this range, the coefficient of friction remains below 0.20. As determined by testing the films are usable at temperatures below 500°F, although their wear-lives are shortened and friction values increase to a range of 0.25 to 0.35. In a limited number of tests these films exhibited coefficients of friction and wear-lives in a vacuum of 10⁻⁶ torr similar to those obtained in air.

Work is continuing at Midwest Research Institute on the development of fluoride-based solid lubricants with special emphasis on
improving the room temperature performance while retaining the outstanding high-temperature performance.

Like many other high-temperature solid-lubricant films, fluoride materials have certain disadvantages in that they have extremely high curing temperatures, up to 1800° F; this has limited their use to the superalloys. The possibility exists that these materials can be flame-sprayed. If this becomes a reality, then applications should be substantially increased because the surfaces onto which they are applied can be kept cool during the coating process and thus eliminate the need for using only superalloy substrates.

VITROLUBE

Vitrolube is the trade name of a ceramic-bonded solid lubricant developed by the North American Rockwell Corp. (ref. 39) to withstand the stringent load and temperature conditions encountered during the operation of the XB-70A, Mach 3 aircraft. Vitrolube contains molybdenum disulfide and graphite in the ratio of 7:2. The glass (ceramic) binder is a combination of glasses which has not previously been utilized in any known dry lubricant coating. The manufacturer states that all components of the coating, including the binder, possess lubricating properties.

A Vitrolube-bonded solid-lubricant coating has been extensively evaluated under conditions closely representing actual use in aerospace vehicles. Results have shown the material to be greatly superior to other materials when tested at approximately 700° F. The Vitrolube material, however, does have some disadvantages. The coating, as is true of most other bonded lubricant films, does not exhibit good corrosion protection; and completely corrosion-resistant substrates must be used to eliminate the problem. Contamination of the wear surfaces by any fluid is detrimental.

The use of Vitrolube is limited to metals that can withstand the high curing temperature required to fuse the ceramic binder. Although developed specifically for use on the XB-70 airplane, a license has been granted to a solid-lubricant supplier, and the material is now commercially available from the company indicated in chapter 2.

GRAPHITE FLUORIDE

Fusaro and Sliney (ref. 40) at the NASA Lewis Research Center have evaluated a new material as a solid lubricant. This material, graphite fluoride, has been known for some time but had not been extensively evaluated as a lubricating solid.

A preliminary investigation of the graphite fluoride material was
conducted on burnished films of the material. Friction and wear tests were pursued on the NASA pin-on-disk sliding-friction apparatus. For comparative purposes, similar tests were conducted on burnished films of graphite and molybdenum disulfide. Fusaro et al (ref. 40) report that the graphite fluoride exhibited coefficients of friction equal to or superior to those of molybdenum disulfide and/or graphite. The wear-life of the burnished graphite fluoride films was up to six times greater than those obtained with graphite or molybdenum disulfide. They also found that moist atmosphere increased the wear-lives of graphite fluoride films, but that it also increased the coefficients of friction.

For practical application, the graphite fluoride solid lubricant is limited to a temperature of approximately 750° F, which is comparable to the usable range of molybdenum disulfide in air.

**RARE EARTH FLUORIDES AND OXIDES**

Other materials being investigated as possible solid lubricants at Lewis Research Center are the powdered lanthanide trifluorides such as LaF₃ and CeF₃. Sliney found (refs. 41 and 42) that significant reductions in wear were obtained when Inconel 600 and Inconel 750 were lubricated with the aforementioned materials in sliding experiments. He also found that the rare earth oxides were not film formers, but he does indicate that fusion-bonded LaF₃ and CeF₃ coatings containing CaF₂–BaF₂ eutectic or LiF as melting point depressants have possibilities as solid-lubricant coatings.
CHAPTER 4

Other Solid Lubricants

In the previous chapters, only the thin-film bonded solid lubricants have been discussed. In addition to these, there are several other types or forms of solid-lubricant materials which may be of more or of equal importance and which are finding increasingly wide use in many industries. This chapter will briefly deal with a number of these materials.

LUBRICATING SOLIDS AS ADDITIVES

Several solid-lubricant materials are added to oils and greases to improve their lubricity and extreme-pressure characteristics. By far the most frequently used solid lubricants for this purpose are molybdenum disulfide and graphite; Teflon and compounds of lead have also been used. Solid-lubricant additives tend to prevent or delay seizure in places where the film of oil or grease is disrupted by high stress or loss of lubricant for finite time intervals. Lubricants containing these additives are used in large quantities by mechanical and electrical manufacturers, equipment-maintenance and automotive industries, as well as government and military agencies. The total quantity of such lubricants used is not known, but it is large; the U.S. Navy alone utilizes over 100,000 lb each year in aircraft, catapults, and other shipboard machinery. Certain automotive companies specify a barium stearate-MoS₂ lubricant for ball-joint applications. The electrical industry uses filled-grease lubricants in switchgear, home appliances, and in the assembly and run-in of large stators.

The quantity of lubricating solids added to grease and oil lubricants varies from 0.5 percent to as much as 60 percent. For general use, a grease containing 5 percent of the lubricating solid is generally the most effective. The choice of quantity and type of additive required will undoubtedly depend on the application.

Judging from the number of manufacturers of liquid lubricants and the quantities being produced, this is probably one of the largest uses of molybdenum disulfide and graphite. Greases containing Teflon and other additives are also available.
LUBRICATING PLASTICS

Plastic bearings fulfill a need that cannot be satisfied by metal bearings. It is desirable and often necessary to reduce or eliminate the lubrication requirements of bearings, and properly designed nonlubricated plastic bearings can often solve this problem. These plastic bearings can also help eliminate problems resulting from: high rate of wear, lubricant failure, chemical corrosion, stick-slip motion, inaccessibility for lubrication, and high- or low-operating temperatures.

PLASTIC BEARINGS

Plastic bearings can be fabricated in any of the conventional bearing configurations such as journal, thrust, combination thrust and journal, flat strip, or special shape (fig. 3).

In addition, thermoplastic bearings can often be incorporated directly in the molded housing of a conveyor-roll end bell, where the housing and bearing are one piece (fig. 4). These bearing materials can be manufactured by injection molding, compression molding, or machining of basic shapes. The final configuration and selected material will determine the most suitable means of fabrication.

CONSTRUCTION MATERIALS

Self-lubricated plastic bearings usually are fabricated from nylon, acetals, tetrafluoroethylene (TFE), polyimides, and phenolics.

Figure 3.—Bearing configurations available in plastic bearings.
These materials are often compounded or blended with other lubricating materials to improve performance.

Nylon is well known for its low friction and good wear, and performs well with little or no lubrication. It swells slightly when exposed to high humidity; under excessive static loads it tends to take a permanent set. A steel sleeve around a nylon bearing reduces cold flow and improves heat dissipation. Wear is less for hardened shafts with a surface finish of 8 to 16 \( \mu \) in. or finer; chromium plating tends to further reduce wear.

Glass fiber fillers in nylon reduce cold flow and thermal expansion, but increase the coefficient of friction. Friction and wear can be reduced by dry-lubricant fillers such as molybdenum disulfide, graphite, and TFE. Small additions of the MoS\(_2\) fillers may also increase the strength.

Acetals cost less than any of the other plastics and have good strength and stiffness. Dynamic and static coefficients of friction are the same, and there is essentially no stick-slip effect at low speeds or in starting. The coefficient of friction against a steel shaft is comparable to nylon. Because of their relatively low cost and good structural properties, acetals are often used as mechanical parts of housings; for example, if a bearing surface is needed, un-
filled acetics can provide good bearing performance without added parts.

Powdered TFE resin or molybdenum disulfide, when added to these plastics in compounding, serves as a dry lubricant to reduce friction and wear. Glass fiber increases stiffness and reduces thermal expansion. TFE fibers reduce friction and impart a very high degree of resistance to wear.

TFE has the lowest friction of any plastic, and its coefficient of friction decreases with load. This resin is resistant to heat, but, like most plastics, is a poor thermal conductor. Except for the fiber form, it needs fillers to provide resistance to cold flow. In addition, fillers increase the load-carrying capacity and reduce the bearing size requirement, the amount of plastic required, and the cost. Thin TFE liners supported by steel bushings also reduce the amount of plastic required, aid in heat dissipation, and permit the use of assembly clearances usual for metal bushings.

When fillers of glass fiber, graphite, and powdered bronze are blended with TFE resin, the \(PV\) values* are about 10,000 at low speeds and over twice as much at speeds of 1000 ft/min. Some work being done with powdered metal fillers points to still higher \(PV\) values with a helpful increase in thermal conductivity, decreased load deflection, and large reductions in wear.

Still another approach combines the low friction of TFE resins with the stiffness and strength of metal. A composite thin layer of porous bronze sintered to a steel backing impregnated with a mixture of TFE resin and powdered lead is an example. \(PV\) values are about the same as for oil-impregnated sintered bronze. At low speeds, steady loads can go up to 20,000 psi with coefficients of friction of 0.05 to 0.1; at speeds of 1000 ft/min, the coefficient nearly doubles.

Various fibers (such as fiber glass, nylon, cotton) when combined with TFE have an extremely high load-carrying ability. In this form they can support loads up to 40,000 psi. Voids in the fabric and thin layers eliminate the problem of thermal expansion when used with a metal backing. However, because of low thermal conductivity, these bearings are limited to low-speed applications. Another limiting factor results from the method of their construction. These fibers are usually bonded to a backing, and as the temperature goes up the bond tends to weaken, limiting the surface speed to about 100 ft/min.

Phenolic plastic bushings have good resistance to cold flow, high

* The \( PV \) value is the product of load on the projected bearing area (psi) and the surface velocity (ft/min).
strength, and high-service temperature, but have higher friction and poor resistance to abrasion. Nevertheless, wear-life can be increased and coefficient of friction reduced by the use of fillers and lubricating additives such as TFE resin. In one line of molded bushings, three lubricants are added: graphite, molybdenum disulfide, and a viscous oil. Fiber fillers arranged radially serve as wicks for the oil; when the bearing warms up and the lubricant flows along the fibers, it produces a true hydrodynamic bearing. This type of bearing will handle combinations of speed and load comparable to sintered bronze bearings with equal or better wear-life and lower coefficient of friction. The thickness of the wall is at least \( \frac{1}{4} \) in.

Polyimides have the highest PV value of any organic material. PV values as high as 100 000 psi at 100 ft/min have been achieved, although in some cases there has been rapid wear. Polyimides are usable over a wide temperature range of from \(-400^\circ\) to \(+700^\circ\) F. Some may even withstand short exposure to temperature up to 900°F without serious damage. When filled with graphite, the wear rate and friction of the polyimide decrease considerably.

Table IX shows some of the mechanical properties—coefficient of friction, wear factor, PV, and effect of temperature for the bearing materials described.

**DESIGN CONSIDERATIONS**

The satisfactory use of a plastic-based bearing depends primarily on three factors: coefficient of friction, wear resistance, and cost. Friction and wear properties will dictate conditions such as load speed and temperature at which each material can be used effectively.

**PV and Wear**

The successful use of any nonlubricated plastic bearing depends upon its load velocity PV capability and wear rate under different service conditions. Limiting PV defines the maximum load and velocity of a material, ignoring the considerations of wear. The limiting PV can indicate whether or not a bearing material should be considered for a specific application.

Since wear is inevitable in a nonlubricated rubbing contact bearing, the design is concerned with the amount of wear. This wear can be determined using a wear factor \( K \), a proportionality constant relating radial wear \( R \) to load \( P \), velocity \( V \), and time \( T \), in the following equation:

\[
R = KPTV.
\]
## Table IX.—Comparative Performance Data for Self-Lubricating Plastic Bearing Materials

<table>
<thead>
<tr>
<th>Properties</th>
<th>Modified polymers</th>
<th>Unmodified polymers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Nylo</td>
<td>Acetal</td>
</tr>
<tr>
<td>Maximum load, projected area, zero speed, psi</td>
<td>4900</td>
<td>5200</td>
</tr>
<tr>
<td>Speed, max ft/min</td>
<td>200-400</td>
<td>500</td>
</tr>
<tr>
<td>PV for continuous service, 0.005 in. wear in 1000 hr, psi × ft/min</td>
<td>1000</td>
<td>1000</td>
</tr>
<tr>
<td>“Limiting PV” at 100 ft/min, psi × ft/min</td>
<td>4000</td>
<td>3000</td>
</tr>
<tr>
<td>Coefficient friction</td>
<td>0.20-0.40</td>
<td>0.15-0.30</td>
</tr>
<tr>
<td>Wear Factor, K × 10⁻¹⁰, min⁻¹ ft⁻¹-lb⁻¹-hr</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Elastic modulus, bending, psi × 10⁶</td>
<td>0.3</td>
<td>0.4</td>
</tr>
<tr>
<td>Critical temp. at bearing surface, °F</td>
<td>400</td>
<td>300</td>
</tr>
<tr>
<td>Thermal conductivity, Btu/in. × hr × sq ft/°F</td>
<td>1.7</td>
<td>1.6</td>
</tr>
<tr>
<td>Thermal expansion, in./in./°F × 10⁶</td>
<td>55</td>
<td>45</td>
</tr>
<tr>
<td>Resistance to humidity</td>
<td>fair</td>
<td>good</td>
</tr>
<tr>
<td>Resistance to chemicals</td>
<td>good</td>
<td>good</td>
</tr>
<tr>
<td>Density, g/cc</td>
<td>1.2</td>
<td>1.43</td>
</tr>
<tr>
<td>Cost index for base material</td>
<td>1.4</td>
<td>1</td>
</tr>
</tbody>
</table>

1. Continuous operation under 5 lb load.
2. Exceeds limiting PV.
Figure 5, which is based on this equation, is a nomograph that relates $PV$ with unit wear per 1000 hr of operation at various "wear-factor" levels. The designer can determine an allowable $PV$ from this information, if he knows the allowable wear of a given application and the wear factor of a particular bearing material. Conversely, having established a required $PV$ level, unit wear can be reasonably predicted.

**Clearances**

The design for plastic journal bearings requires that proper running clearances on the shaft be specified. Because of the high thermal expansion of most plastics, these bearings require larger clearances than those normally used with metal bearings. In some cases, the possible disadvantages of these large clearances are overcome by the resilience and vibration-dampening effects of the plastic. The recommended clearance for most nonlubricated plastic bearings is 0.005 in./in. of shaft diam for solid journal bearings. If a split-curl configuration is used to eliminate seizing, clearances can be reduced to 0.001 in./in.

**Shaft Finish**

Most plastic bearings can be used either as molded or machined, as the surface finish on the plastic bearing is not critical. The mating shaft should have a finish of 8 to 16 $\mu$ in. rms for best performance. Too smooth a surface is unnecessarily expensive and
rougher surfaces tend to abrade the bearing. The effect of shaft finish on the wear of TFE bearings is shown in figure 6.

**COST CONSIDERATIONS**

Cost is, of course, an important consideration in the selection of bearing material. On a raw material basis the acetals are the least costly, followed by nylon, phenolics, fluorocarbons, and polyimides. However, raw material costs do not necessarily give a good indication of the cost of finished parts. For instance, when using nylon and acetals it may be possible to incorporate the bearing directly in the injection-molded housing, eliminating the need for a separate piece and an assembly operation. Fluorocarbon bearings are often fabricated in a thin tape that is inserted in a housing at very low cost compared to a solid-machined bearing. At the present time, the polyimide bearing is expensive, but if the designer is faced with a severe nonlubricated bearing problem it will usually pay for itself. In any plastic bearing design the real criterion is value in use. Practically every industry has used or can use nonlubricated plastic bearings. Some typical applications are included as figures 7 through 17.

**POWDERED SOLID LUBRICANTS**

The simplest and oldest form of solid lubrication, still widely used, merely involves applying lubricating solids to the surfaces.
FIGURE 7.—Spherical bearings lined with fabrics of Teflon.

FIGURE 8.—Bearing retainers of Delrin acetal resin.

FIGURE 9.—Ball bearing retainers of filled Teflon resin.

FIGURE 10.—Bearing of Teflon resin.
FIGURE 11.—Self-aligning bearing of Zytel resin.

FIGURE 12.—Self-aligning bearing and housing of Delrin AF fiber/resin and Zytel resin, respectively.

FIGURE 13.—Elevator gib of Zytel resin.

FIGURE 14.—Self-aligning bearings of Zytel resin.
OTHER SOLID LUBRICANTS

FIGURE 15.—Manure spreader bushings of Teflon resin.

FIGURE 16.—Glide pads of Teflon resin.

FIGURE 17.—Door-pivot bearings of glass-filled tape of Teflon resin.
requiring lubrication. A wide variety of powdered solids can be used. In this method, the lubricating film is formed on the bearing or wearing surface by merely covering the surface with a finely divided lubricant powder. The resultant film reduces both friction and wear of the mating surfaces. Bonding to the metal substrate depends upon intimate contact between the lubricant powder and metal substrate and their affinity for each other. The surface roughness of the substrate may also create small reservoirs and trap the loose powder which produces some increase in adhesion.

Methods of application include rubbing, burnishing, dusting, tumbling, and aerosol spraying.

*Rubbing.*—Distributing the lubricant powder over the bearing area by rubbing with the hands is a common method. This usually produces a very loose coating, but is effective in the assembly of close tolerance parts.

*Burnishing.*—This procedure is the same as for rubbing except a greater force is applied, usually by some mechanical means. The greater load will compact more lubricant into the surface imperfections and usually result in a longer lasting film. After burnishing, the coating will be smooth and glossy. This method is suitable for parts whose wear-lives are not critically important.

*Dusting.*—In this method, no attempt is made to distribute the powder evenly, resulting in an uneven and generally unsatisfactory coating.

*Tumbling.*—Coating is accomplished by mixing normal tumbling media (such as balls, spheres, cones, and needles) with dry powder. Parts to be coated are then placed in this mixture and tumbled for a period of time. Although adhesion is not good in this method, it is suitable for coating noncritical items such as small threaded fasteners and rivets. Care must be taken when using a dry lubricant on threaded fasteners because the accepted dry-torque levels are not applicable.

*Aerosol Spraying.*—In this method, a finely divided lubricant is applied using an aerosol spray with a volatile carrier such as alcohol or Freon. Again, adhesion is not good, but the ease of application may offset this disadvantage.

The solid-lubricant powders that can be used by these application methods include molybdenum disulfide, graphite, mica, Teflon, talc, and tetrafluoroethylene. These powders can be used in forming, press-fitting, and wire-drawing operations; and with fasteners (threaded and rivet) and dies.

The use of the powdered materials is usually limited to areas where adhesion of the lubricant is not critical. Many of the methods of application discussed in previous chapters were developed as a
direct result of attempts to obtain better bonding of the materials in the powdered form. The prime requirements for such a material are low coefficient of friction and the ability to prevent wear.

**GAS-ENTRAINED POWDERS**

A component (usually antifriction bearings) can be lubricated by a solid-lubricating powder carried to the bearing by a stream of gas. The gases employed are usually inert (e.g., argon or nitrogen), since this method is intended for high-temperature application. The inert carrier gas helps to prevent oxidation of the lubricating solids. Most work in this area has been on an experimental basis. This method has not been used in any known system, but several investigators (refs. 43 through 46) have studied its feasibility in lubricating high-speed bearings.

A problem that can be anticipated with this method of lubrication is bearing fatigue. This phenomenon has been studied to some extent by Carter (ref. 47) using MoS₂ carried in a liquid. At temperatures where the liquid evaporated, there was much lower fatigue life, thought to be caused by stress raisers formed by the solids in the region of pure-rolling contact.

**LUBRICANT COMPOSITES**

The lubricant composites to be discussed here are mixtures of lubricating and metal powders compacted under high load at elevated temperatures to form a solid body containing a high percentage of lubricating material. A solid lubricant is transferred from within the structure of the composite to the interface between the moving parts, forming an effective lubricating film.

In 1950, personnel at the Lewis Flight Propulsion Laboratory (now Lewis Research Center) investigated the feasibility of lubricating bearings in this manner. They also established the amount of lubricant necessary for such a bearing material. An investigation of a composition containing MoS₂, silver, and copper (ref. 48) indicated that effective lubrication could be obtained with a material containing 10 percent MoS₂, 5 percent copper, and 85 percent silver, and that this material gave the lowest rate of wear with a relatively low-friction value. The friction appeared to be unaffected by sliding velocity. Welding of the surfaces occurred with composites containing less than 5 percent of the MoS₂ lubricating powder.

Other investigators have used this method of lubricating ball and roller bearings. Campbell and Van Wyk (refs. 49 and 50) reported on the development of a lubricant composite in which they were
attempting to lubricate 204-size bearings operating in air and in a vacuum of $10^{-6}$ torr at 1500° F. Their report mentions between 400 and 500 different material combinations that were fabricated during their investigation. Unlike the Lewis investigators, they used extremely high percentages of lubricating powders (e.g., MoS$_2$ and PbO) and hard metal binders, rather than silver. In this work, the two best materials were reported to be 90 percent MoS$_2$, 8 percent Fe, and 2 percent Pt; and 90 percent MoS$_2$, 8 percent Fe, and 2 percent Pd. Operation of the 204-size bearings was achieved at speeds to 15,000 rpm in a vacuum of $10^{-6}$ torr, at 700° F. The lubricant composite supplied enough lubricant to the moving surfaces so that a very thin, highly burnished lubricant film was maintained. This work demonstrated also that lubricant composite materials can be made with sufficient strength to warrant their use as cage materials for bearings.

Composite development at The Boeing Co. has been continued with later work being reported by Hubbell et al (ref. 51). Bowen et al (refs. 52 through 55) of the Westinghouse Electric Corp. also have done considerable developmental work on lubricant composites. They have included such lubricant powders as polytetrafluoroethylene, graphite, antimony sulfide (Sb$_2$S$_3$), iron sulfide (FeS), selenides and tellurides of tungsten, and molybdenum. Work on these programs has been mainly on the lubrication of gears and related equipment. Other investigators, including Devine (ref. 56), have compacted the common solid-film lubricants into solid sections for use as bearing components.

One problem usually associated with a new idea or product, such as metal-bonded composites, is transferring it from research to practical use. At least one company has taken the initiative in this area and is now offering several of the Boeing-developed composite materials for sale (ref. 57). The metal-bonded lubricants have been used successfully in the field of electrical brushes and contacts, particularly in vacuum applications.

Ulrich (ref. 58) has summarized work completed on hot-pressed MoS$_2$-Ag electric contact motor brushes. He stated that compositions of hot-pressed MoS$_2$ containing metallic additives have shown promise as materials for electric contact brushes in vacuum. He further stated that the brushes studied have properties of degenerated semiconductors and that the analysis conducted indicated that the best combination would be a mixture of 80.5 percent MoS$_2$-19.5 percent silver. Testing of brushes of the best combination appears to confirm the results of the analysis.

Demorest and Whitaker (ref. 59) investigated new brush materials for low wear and good electric contact by evaluating the ma-
terials in dc motors running in vacuum. They found several materials that gave reasonably good results, including NbSe$_2$ (niobium diselenide), NbSe$_2$ + 20 percent MoS$_2$, TaS$_2$ (tantalum disulfide) + graphite + NbSe$_2$, and WSe$_2$ + silver, but certain difficulties were encountered with each of these formulations. The NbSe$_2$ and NbSe$_2$ + MoS$_2$ brushes were brittle and chipped badly. The TaS$_2$ + graphite + NbSe$_2$ brushes caused severe wear on the commutator, and the WSe$_2$ + silver brushes wore severely during a relatively short test.

The best results were obtained by Demorest and Whitaker (ref. 59) when they used, for a brush material, a metal-bonded composition that had been fabricated specifically for a cage material in rolling-element bearings. This material contains molybdenum disulfide, molybdenum, and tantalum (manufacturers' designation 046-45). Some typical properties of this material are given in table X. In motor tests under vacuum conditions the 046-45 brush mate-

<table>
<thead>
<tr>
<th>Density</th>
<th>5.6 g/cc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexible strength</td>
<td>12 480 lb/in.$^2$</td>
</tr>
<tr>
<td>Compressive strength</td>
<td>49 000 lb/in.$^2$</td>
</tr>
<tr>
<td>Shear strength</td>
<td>8000 lb/in.$^2$</td>
</tr>
<tr>
<td>Composition</td>
<td>(proprietary) contains MoS$_2$, Mo, and Ta</td>
</tr>
</tbody>
</table>

rial was found to have a wear rate of approximately $10^{-5}$ in./hr. These brushes caused little wear on the commutator and little or no arcing. It appears that the 046-45 brush material does have a slightly higher wear rate when tested in air, on the order of $10^{-4}$ in./hr.

Additional work is being done at Marshall Space Flight Center on electric brush material. Other investigations have been or are being conducted on electric brushes composed of metal and lubricant powders by personnel at Goddard and Marshall Space Flight Centers (refs. 60 through 62) and at the Lockheed Missiles and Space Co. (now Lockheed Aircraft Corp.) (refs. 63 and 64).

**SOLID-LUBRICANT LAMINATES**

Laminate materials have been developed which are of some interest because of their relatively high-bearing strength (10 000 to 38 000 psi) and hardness ($R_m > 100$). Generally, these materials are made of layers of paper, cloth, fiber glass, or nylon fabric with resin and MoS$_2$, graphite, or Teflon compressed under pressure and heat. $PV$
values for these materials range from 10,000 at 10 ft/min to 30,000 at 1000 ft/min, and the wear factor $K$ ranges from 15 to 40 in.$^3$ min/ft. lb-hr.

The coefficient of friction of laminate materials is relatively high for lubricating solids, ranging from 0.17 to 0.21. The temperature ranges for these materials are limited by the fabric and resin employed, which generally restrict them to a maximum temperature of 400° F.
Methods of Evaluating Solid Lubricants

Methods of evaluating solid-film dry lubricants are numerous. Nearly every major user of bonded solid films has his own particular test machine for evaluating the wear-life and obtaining the coefficient of friction of materials over the range of conditions that he feels is important. In addition to the wear-life and friction tests, there are minor tests which are nearly standard throughout industry. These minor tests include film adhesion, fluid resistance, heat resistance, and corrosion resistance. Most of the minor tests are fully described in the applicable specifications, Federal test method standards, or ASTM test method standards.

Such a wide variation in test results on film life has been reported by various organizations, that the Coordinating Research Council (CRC) conducted a round-robin test program in an effort to determine the correlation, if any, between two of the most widely accepted test machines, the Falex and MacMillan. The conclusions drawn showed no correlation between these two machines. Also, very little repeatability existed between testers of the same machine in different laboratories. The CRC did find that more repeatability existed between laboratories using the Falex tester than those using the MacMillan tester. On the basis of these findings, the government agency having cognizance over the solid-lubricant specifications switched to the Falex machine and now requires its use in all solid-film lubricant qualification testing.

The American Society of Lubrication Engineers' Subcommittee on Wear of the Lubrication Fundamentals Committee (ref. 65) has compiled information on over 100 test machines used in the evaluation of lubricants. Devine (ref. 66) has compiled a list of test machines (table XI) frequently used in the evaluation of solid lubricants.

Most of the test data on solid dry lubricants result from the measurement of the friction and wear characteristics in bench-type laboratory tests on full-scale bearings. Some chemical tests have been used for evaluating solid-lubricant properties, but these serve more as specification controls than as developmental tools. Because no single organization has attempted to design and construct a standard test machine for dry lubricants, the industry has relied
### Table XI.—Examples of Friction and Wear Test Apparatus for Solid Lubricants

<table>
<thead>
<tr>
<th>Name</th>
<th>Conditions</th>
<th>Specimen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plain Bearing Tester</td>
<td>1. 630°F</td>
<td>H–11 test shaft, M–2 steel bushing</td>
</tr>
<tr>
<td></td>
<td>2. 10 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. ±32 deg/cycle</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. 10,000 psi</td>
<td></td>
</tr>
<tr>
<td>Friction Tester</td>
<td>1. 77°F</td>
<td>Metal disk and rider</td>
</tr>
<tr>
<td></td>
<td>2. 65 cm/sec</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Variable load</td>
<td></td>
</tr>
<tr>
<td>High-Temperature,</td>
<td>1. 750°F</td>
<td>20-mm rolling-contact bearing</td>
</tr>
<tr>
<td>High-Speed Bearing</td>
<td>2. 10,000 rpm</td>
<td></td>
</tr>
<tr>
<td>Performance Apparatus</td>
<td>3. 5-lb thrust load</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3-lb radial load</td>
<td></td>
</tr>
<tr>
<td>Vacuum Friction and</td>
<td>1. 77°F</td>
<td>Metal disk and rider</td>
</tr>
<tr>
<td>Wear Apparatus</td>
<td>2. 390 ft/min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 1000 g</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. 10−6 to 10−7 mm Hg</td>
<td></td>
</tr>
<tr>
<td>Lubricant Vacuum</td>
<td>1. 77°F to 1000°F</td>
<td>20-mm rolling-contact bearing</td>
</tr>
<tr>
<td>Environment Apparatus</td>
<td>2. 1250 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 5-lb thrust load</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3-lb radial load</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. 1x10−6 mm Hg</td>
<td></td>
</tr>
<tr>
<td>Modified 4-Ball Wear Machine</td>
<td>1. 1000°F</td>
<td>½-in. diam balls</td>
</tr>
<tr>
<td></td>
<td>2. 200–700 ft/min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 2–8 kg</td>
<td></td>
</tr>
<tr>
<td>High-Temperature,</td>
<td>1. 1000°F to 1800°F</td>
<td>Rotating washer vs stationary pad stators</td>
</tr>
<tr>
<td>High-Speed Rubbing Wear</td>
<td>2. 100–200 ft/min</td>
<td></td>
</tr>
<tr>
<td>Apparatus</td>
<td>3. 5–50 psi</td>
<td>Pellet vs tool steel track</td>
</tr>
<tr>
<td>Pellet Friction Machine</td>
<td>1. 80°F to 1000°F</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2. 600 ft/min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 128–326 psi</td>
<td></td>
</tr>
<tr>
<td>Press-Fit Tester</td>
<td>1. 77°F</td>
<td>Pin and bushing</td>
</tr>
<tr>
<td></td>
<td>2. 0.6–2 in./min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Variable load</td>
<td></td>
</tr>
<tr>
<td>Lubricant Tester</td>
<td>1. 77°F</td>
<td>Test block and 1½ in. Timken bearing* outer race</td>
</tr>
<tr>
<td>Model LFW–1</td>
<td>2. 12–197 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 30–630 lb</td>
<td>Cylindrical pin and V-blocks</td>
</tr>
<tr>
<td>Falex Lubricant</td>
<td>1. 77°F</td>
<td>Cylindrical cup rotating against a stationary rectangular block</td>
</tr>
<tr>
<td>Tester</td>
<td>2. 290 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 200–2500 lb (gauge)</td>
<td></td>
</tr>
<tr>
<td>Timken Tester</td>
<td>1. 77°F</td>
<td>Rotating ring vs stationary blocks</td>
</tr>
<tr>
<td></td>
<td>2. 200–1300 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 6300–50,400 psi</td>
<td></td>
</tr>
<tr>
<td>Oscillating Tester</td>
<td>1. 400°F</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2. 230 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 62 deg arc</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. 700 lb</td>
<td></td>
</tr>
<tr>
<td>Name</td>
<td>Conditions</td>
<td>Specimen</td>
</tr>
<tr>
<td>----------------------------------</td>
<td>-----------------------------------</td>
<td>-----------------------------------</td>
</tr>
<tr>
<td>Dry-Film Lubricant Tester</td>
<td>1. 100°F to 1700°F</td>
<td>Cylindrical pin and V-blocks</td>
</tr>
<tr>
<td></td>
<td>2. 3.5 ft/min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 300 lb</td>
<td></td>
</tr>
<tr>
<td>Sliding Friction Test Apparatus</td>
<td>1. 77°F to 300°F</td>
<td>Cylindrical sleeve</td>
</tr>
<tr>
<td></td>
<td>2. 47 slides/hr</td>
<td>slider sliding on</td>
</tr>
<tr>
<td></td>
<td>3. Oscillated ±48 deg</td>
<td>a cylindrical rod</td>
</tr>
<tr>
<td></td>
<td>4. $10^{-6}$–$10^{-9}$ torr</td>
<td></td>
</tr>
<tr>
<td>Bearing Test Apparatus</td>
<td>1. 77°F</td>
<td>20-mm rolling-contact bearing</td>
</tr>
<tr>
<td></td>
<td>2. 1800 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. $\frac{1}{2}$ lb axial and</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\frac{1}{2}$ lb radial</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. $10^{-7}$–$10^{-8}$ torr</td>
<td></td>
</tr>
<tr>
<td>Fretting Corrosion Apparatus</td>
<td>1. 77°F</td>
<td>Steel flat vibrating against steel flat</td>
</tr>
<tr>
<td>High-Temperature Bearing Radial and Thrust Rig</td>
<td>1. 1200°F</td>
<td>20-mm rolling-contact bearing</td>
</tr>
<tr>
<td></td>
<td>2. 20,000–35,000 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 50-lb thrust and/or</td>
<td></td>
</tr>
<tr>
<td></td>
<td>50-lb radial</td>
<td></td>
</tr>
<tr>
<td>Modified Lubricant Tester</td>
<td>1. 77°F</td>
<td>Rotating ring vs rectangular block</td>
</tr>
<tr>
<td></td>
<td>2. 72 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 630 lb</td>
<td></td>
</tr>
<tr>
<td>Modified Falex Tester</td>
<td>1. $-321^\circ$ F ($-196^\circ$ C)</td>
<td>Rotating pin vs stationary V-blocks</td>
</tr>
<tr>
<td></td>
<td>2. 290 rpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 1000 lb (gauge)</td>
<td></td>
</tr>
<tr>
<td>Crossed Cylinders Friction Apparatus</td>
<td>1. 77°F</td>
<td>Vertical cylinder—carbon tool steel, horizontal cylinder—solid section</td>
</tr>
<tr>
<td></td>
<td>2. 8–367 sfm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 1–5 lb</td>
<td></td>
</tr>
<tr>
<td>Model A–3 Friction and Wear Tester</td>
<td>1. 77°F</td>
<td>Timken test cup</td>
</tr>
<tr>
<td></td>
<td>2. 72 rpm</td>
<td>T-54148 and AISI-4130 steel blocks</td>
</tr>
<tr>
<td></td>
<td>3. 630 lb</td>
<td></td>
</tr>
<tr>
<td>Stick-Slip Slow-Speed Friction Apparatus</td>
<td>1. 80°F to 1000°F</td>
<td>$\frac{1}{2}$ in. diam ball vs flat block</td>
</tr>
<tr>
<td></td>
<td>2. 0.17 mm/sec</td>
<td></td>
</tr>
<tr>
<td>Rolling and Sliding Contact Specimen Test Machine</td>
<td>1. $-100^\circ$ to $+2000^\circ$ F</td>
<td>Ball and plate</td>
</tr>
<tr>
<td></td>
<td>2. 22 cpm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Duration—30 min</td>
<td>Rotating ring vs rectangular blocks</td>
</tr>
<tr>
<td>A–6 Friction and Wear Tester</td>
<td>1. $-60^\circ$ to $+1500^\circ$ F</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2. 200 ft/min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. 16,000 psi</td>
<td></td>
</tr>
<tr>
<td>Gallling Machine</td>
<td>1. 80 to 900°F</td>
<td>Flat plate vs test cup</td>
</tr>
<tr>
<td></td>
<td>2. 3.5–300 ft/min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Up to 25,000 psi</td>
<td></td>
</tr>
</tbody>
</table>

Notes:
(*) Timken: registered trademark.
(*) RT: Room temperature.
(*) sfm: surface ft/min
heavily on equipment used to evaluate liquid lubricants. The lack of standard methods of evaluation has created problems in comparing the results of various tests. Additionally, many of the solid lubricants do not lend themselves to standardization of test methods.

**FRICTION AND WEAR TESTERS**

Although a wide variety of friction and wear testers have been used for the evaluation of solid lubricants, only the most common test machines (table XII) will be discussed in this chapter.

**Falex Lubricant Tester**

Since the organization controlling the dry-lubricant specifications is using the Falex test machine (fig. 18) exclusively in its test work, it will be discussed first. A rotating pin is held between two stationary V-blocks in a nutcracker-type loading system (fig. 19). This tester gives initial line contact on loading, with eventual area contact as wearing takes place. Although this machine has been used mainly in the evaluation of thin-film solid lubricants at room temperature, it has been modified for testing at both liquid nitrogen
<table>
<thead>
<tr>
<th>Name</th>
<th>Specimen geometry</th>
<th>Load range variable</th>
<th>Environment</th>
<th>Temperature, °F</th>
<th>Figure No.</th>
<th>Reference No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Falex Lubricant Tester ¹</td>
<td>Pin between two V-blocks</td>
<td>4500 lb</td>
<td>Air</td>
<td>Ambient</td>
<td>18</td>
<td>70</td>
</tr>
<tr>
<td>LFW-1 ²</td>
<td>One block on rotating disk</td>
<td>600 lb</td>
<td>Air</td>
<td>Ambient</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td>LFW-3 ²</td>
<td>Annular ring on flat block</td>
<td>100–5000 lb</td>
<td>Air</td>
<td>Ambient to 1200</td>
<td>35</td>
<td>72</td>
</tr>
<tr>
<td>LFW-4 ²</td>
<td>Pin and bushing</td>
<td>12,000 lb</td>
<td>Air</td>
<td>Ambient</td>
<td>36, 37</td>
<td>73</td>
</tr>
<tr>
<td>LFW-5 ²</td>
<td>Journal and shaft</td>
<td>210 lb</td>
<td>Air</td>
<td>Ambient to 750</td>
<td>45</td>
<td>75</td>
</tr>
<tr>
<td>Hohman A-6 ³</td>
<td>Two blocks on rotating disk</td>
<td>600 lb</td>
<td>Air</td>
<td>Ambient to 1600</td>
<td>24, 25</td>
<td></td>
</tr>
<tr>
<td>NASA High-Load Tester ⁴</td>
<td>Flat on flat</td>
<td>150,000 lb</td>
<td>Air</td>
<td>Ambient</td>
<td>38, 39</td>
<td>74</td>
</tr>
<tr>
<td>NASA High-Temperature Friction Apparatus ⁵</td>
<td>3/16 in. hemispherical rider on rotating flat disk</td>
<td>Variable</td>
<td>Air and Vacuum</td>
<td>Cryogenic to 2000</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>MARK VB ⁶</td>
<td>Two blocks on rotating disk</td>
<td>600 lb</td>
<td>Air and Vacuum</td>
<td>Ambient to 1500</td>
<td>26, 27</td>
<td>71</td>
</tr>
<tr>
<td>Pellet Wear-Life Apparatus ⁷</td>
<td>Three pellets on flat plate</td>
<td>800 g</td>
<td>Vacuum, air, and dry N₂</td>
<td>-100 to +450</td>
<td>28, 29, 30</td>
<td></td>
</tr>
<tr>
<td>Oscillatory-Bearing Tester ⁶</td>
<td>Plain spherical bearing</td>
<td>3500 lb</td>
<td>Air</td>
<td>Ambient to 1000</td>
<td>33, 34</td>
<td></td>
</tr>
<tr>
<td>Journal-Bearing Tester ⁶</td>
<td>Plain spherical bearing</td>
<td>5000 lb</td>
<td>Air</td>
<td>Ambient</td>
<td>41, 42, 43, 44</td>
<td></td>
</tr>
<tr>
<td>Oscillatory-Sliding Friction and Wear Tester ⁶</td>
<td>Flat on flat</td>
<td>1500 lb</td>
<td>Air</td>
<td>Ambient</td>
<td>47, 48</td>
<td></td>
</tr>
</tbody>
</table>

¹ Faville-LeVally Corp., Bellwood, Ill.
² Dow Corning Corp., Stamford, Conn.
³ Hohman Plating and Manufacturing Inc., Dayton, Ohio
⁵ NASA—Lewis Research Center, Cleveland, O.
⁶ Midwest Research Institute, Kansas City, Mo.
⁷ Bearing locked to shaft to cause rotation on spherical surface.
and elevated temperatures (refs. 67 through 69). A more complete description of the test machine and procedure is given in reference 70.

**Rub-Shoe Testers**

Another extensively used test for the evaluation of solid lubricants has been the flat block, or shoe, bearing on a rotating ring or disk as shown schematically in figure 20. A similar arrangement is shown in figure 21, but with diametrically opposed rub shoes. Either version gives initial line contact, with area contact resulting from load and wear. These testers are normally employed for evaluating resin or ceramic-bonded films.

**LFW-1.**—This machine uses the test configuration shown in figure 22, where a stationary test block is pressed against a rotating Timken ring. The rotational speed varies from 12.5 to 197 rpm, and the load from 30 to 630 lb. Testing is done at room temperature only, friction being monitored throughout the test period. The machine, which can be operated in both unidirectional and oscillatory motion, gives initial line contact on loading, with area contact resulting from load and wear. The test machine and related equipment is shown in figure 23.

**Hohman A-6*.**—The Hohman A-6 tester uses diametrically opposed blocks as shown in figure 21, the load varying to 600 lb. The

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* Hohman Plating and Manufacturing, Inc., Dayton, O.
machine can operate to 1600° F. As with the Falex and LFW-1 machines, initial line contact is obtained on loading, with area contact resulting from wear and load. A schematic drawing of the test configuration is shown in figure 24 and a complete tester is shown in figure 25.
MRI Mark-VB rub-shoe machine.—Like the Hohman A-6 machine, the Mark-VB is a rub-shoe machine with diametrically opposed shoes. A schematic drawing (fig. 26) shows the test configuration and loading system. The capacities of this machine (fig. 27) are: 600 lb on each shoe, variable speed of 100 to 1000 rpm (continuously variable), operation at room temperature to 1500° F, and operation at vacuum of 10⁻⁶ torr. A more complete description can be found in reference 71.
Other Testers

MRI pellet wear-life apparatus.—This small tester was developed by MRI for NASA. A schematic test configuration and photographs of the apparatus are shown in figures 28, 29, and 30. This apparatus is intended for use in conducting, primarily, wear-life investigations at light loads; it operates unattended for extended periods of
time. The specimen configuration is three \( \frac{1}{4} \) in.-diam pellets sliding over a flat plate. The unit has the following capacities: load 300 g/contact, speed to 765 ft/min, and operation at room temperature in a normal atmosphere.

*MRI vacuum friction apparatus.*—This equipment uses the same specimen configuration shown in figure 28, but it is designed for
FIGURE 29.—MRI pellet wear-life tester (cutaway).

FIGURE 30.—MRI pellet wear-life test machine.
operation at a vacuum of $10^{-7}$ torr. Figure 31 shows the drive mechanism, and figure 32 shows the entire machine. Additional information on this apparatus is available in reference 28.

**MRI oscillatory-bearing tester.**—The capacities of this tester are: 10,000-lb load, operation from room temperature to $1000^\circ$F, and oscillatory motion of ±15 at 9 cpm. Specimens tested in this machine are 5/6-in. bore plain spherical bearings. The tester and specimen configurations are shown in figures 33 and 34. On this tester, the spherical bearing is locked on the shaft causing the bearing to operate on the spherical area.

**LFW-3.**—The capacities of this machine are: operation from room temperature to $1200^\circ$F, 100 to 5000-lb load, rotational speed from 9 to 320 rpm, and oscillatory frequency of from 6 to 227 cpm ($0^\circ$ to $60^\circ$, 6 to 227 cpm; $0^\circ$ to $120^\circ$, 6 to 120 cpm). Test specimens are of the annular ring variety with a constant theoretical area of contact (fig. 35). Additional information on this machine is available in reference 72.

**LFW-4—press-fit testing machine.**—This is a new testing machine for boundary friction studies. It utilizes the simple pin and bushing specimens shown in figure 36. The machine is intended for studies on static and kinetic friction during and after "wear-in" galling, seizing and the problem of stick-slip (fig. 37). Additional information on this machine can be obtained from reference 73.

**NASA high-load tester.**—A photograph of this test device is shown in figure 38 and a schematic in figure 39. It consists of a
FIGURE 32.—MRI vacuum friction apparatus.

FIGURE 33.—MRI oscillatory-bearing test machine.
frame on which is mounted a 50-ton mechanical jack that applies a normal load to the specimen through a load cell. Test specimens consist of two self-aligning plates which fit into machined recesses in the frame and load-cell support plate. The slider shown in figure 40 is positioned between the self-aligning plates and is connected through a load cell to the drive mechanism. Unit loads of 150,000 psi on projected areas of 0.59 sq in. can be applied by this tester. Its speed is low, 0.67 in./min, with a total travel of $\frac{1}{2}$ in. in each
direction. Additional information on this tester is available in reference 74.

NASA friction and wear machine.—Figure 2 showed the arrangement of this machine and gives a close-up of the test specimen and hemispherical rider. Basically, the apparatus consists of a rotating disk placed in sliding contact with a hemispherically tipped rider, usually under a 1000 g load. Sliding is unidirectional at controlled speeds up to 1260 ft/min. A more complete description of this type machine can be found in references 40 and 41.
**MRI journal-bearing tester.**—This tester (fig. 41) is used to measure the coefficient of friction and wear-life of bonded solid lubricants applied to plain, spherical journal bearings operating on cylindrical shafts. The test shafts are hardened dowel pins chucked in two precision collets that are mounted in pillow blocks. The test journal is the ball of a standard ⅜-in. diam spherical bearing (fig. 42), the spherical surface of which is used only for initial alignment and is not lubricated. The replacement of the journal bearing is facilitated by using a loader-slot spherical bearing seat. The load is applied to the journal by a 5-in. bore pneumatic cylinder (fig. 43) through a hanger in which the spherical bearing seat is mounted. Regulated bottled gas is used to control the load on the journal bearing and the load hanger is instrumented with semiconductor strain gauges for the measurement of both load and torque (fig. 44).

The test shaft is driven through one of the collets by an SCR-controlled dc motor with an integral gear reducer, shaft speeds being adjustable from 5 to 125 rpm. Modified versions of these journal-bearing testers are operated at speeds up to 3600 rpm. Wear-life of the journal bearing is measured by a running-time meter on the motor controller, and the torque, sensed by the strain gauges, is indicated on a meter relay and registered on an external recorder (fig. 43). The meter relay will turn off the drive motor when a preset torque is exceeded.
LFW-5 journal-bearing tester.—The LFW-5 journal-bearing tester (fig. 45) uses the standard test shaft and bushing shown in figure 46. Unlike the MRI machine, the journal is not spherical and is consequently difficult to align. It is available in two versions: (1) a standard model with fixed speeds of 1000 or 3000 rpm and (2) a variable speed model that can operate from 0 to 5000 rpm. The tester is fully automatic and can be operated from ambient to 750°F (ref. 75).

MRI oscillatory-sliding friction and wear machine.—The MRI sliding-friction and wear tester shown in figure 47 uses a flat-plate-on-flat-plate test configuration shown in figure 48. A deadweight and lever system applies a load, adjustable from 150 to 1500 lb, to the upper section. The contact area is adjustable 0.1 to 1.0 sq in. and contact stresses can be varied from 150 to 15,000 psi. The lower specimen holder is a wear plate 1½ in. by 6 in., driven by a crank, pitman arm, and slider, to provide a nearly sinusoidal reciprocating
FIGURE 40.—NASA high-load tester slider.

FIGURE 41.—MRI journal-bearing machines.
FIGURE 42.—Journal-bearing test configuration.

FIGURE 43.—Journal testers—controls and load cylinder.

FIGURE 44.—Journal tester—strain gauges and drive collets.
**Figure 45.**—LFW-5 test machine.

**Figure 46.**—LFW-5 test configuration.
motion. The speed is adjustable from 2 to 60 strokes/min, and the stroke is adjustable from 1 to 4 in. in \( \frac{1}{2} \)-in. increments. The frictional force is measured by a strain gauge link in the constraint linkage of the upper specimen holder.

Frictional force is recorded on a strip chart recorder-controller
with a set-point control used to shut off all power to the tester when
the friction exceeds a predetermined value. The running time is
indicated automatically.

Although many testers are available for the evaluation of bonded
solid lubricants, the results from any one tester cannot necessarily
be taken as the absolute answer. To demonstrate the point more
clearly, an analysis of contact stresses found in three of the most
widely used machines is included as appendix B. It is suggested
that the above analysis be studied thoroughly before deciding on a
particular test configuration.
CHAPTER 6

Effects of Environments on Solid Lubricants

Solid-lubricant materials are generally selected for use because of their resistance to environmental conditions that conventional oil and grease cannot tolerate. Other advantages solid lubricants have over conventional lubricants include (1) lubrication for the life of the part, (2) essentially dry lubrication, (3) lubrication of parts of components that are difficult to reach, (4) lubrication where the presence of vapors from fluid or grease lubricants is unacceptable, and (5) no attraction for dust or dirt.

Environmental conditions can strongly influence the performance of solid lubricants. Friction and wear characteristics may vary from one machine element to another using the same lubricant. The reader, however, is cautioned that most solid-lubricant data are based on laboratory and bench tests and that such values do not necessarily correlate directly with those obtained from operational experience. Therefore, the data should be considered only as guides. It is strongly suggested that those interested in using solid lubricants should conduct evaluation tests on equipment known to correlate with their particular requirement. If this type of testing had been conducted in the early days of dry-film lubricants, many misapplications would have been avoided. Excessive reliance on manufacturers' bench-test data has resulted in severe criticism and a general distrust of all solid lubricants. A solid lubricant, more than other lubricants, requires careful consideration of the operating conditions to which it is subjected.

EFFECTS OF NORMAL ATMOSPHERE

From the research presently being conducted and the volumes of literature available on solid lubricants, it appears that very little work has been done on solid lubricants under the more conventional conditions—normal air environment at temperatures in the range of \(-65^\circ\) to \(+200^\circ\) F. On the other hand, nearly all research reported is for very specific conditions. Apparently, the feeling has been that since there are specifications covering the solid lubricants at normal conditions, there is no need for further work in the area. Formerly, no attempt was made to arrange the known characteristics of solid
lubricants in normal air atmosphere into a usable document for designers.

In the normal atmosphere, the solid lubricants come into contact with such contaminants as dust, dirt, water vapor, salt water, oil, and other lubricants, all of which adversely affect their usefulness. Water vapor or humidity is a good example of these contaminants. Several investigators have shown that water vapor causes molybdenum disulfide solid lubricants to have higher coefficients of friction, while the reverse is true of graphite which depends on a certain amount of moisture to permit it to act as a lubricant.

Water vapor can also cause other problems, the major one being corrosion. The thin-film bonded lubricants of the resin-, nonceramic-, and ceramic-bonded types are all susceptible to damage from substrate corrosion. Water can enter the pores and react with the base material or substrate, forming rust or other corrosion products. The situation is further aggravated by the presence of a salt solution. Researchers at the U.S. Army Rock Island Arsenal (refs. 76 and 77) have conducted extensive corrosion studies on resin-bonded solid lubricants. They found that up to 50 percent (area coverage) corrosion has little effect on wear-life, but that complete rust coverage reduces the wear-life otherwise expected to approximately one-half. They also found that conventional lubricants are harmful to the wear-life of a bonded solid-film lubricant.

The MRI group has conducted a corrosion study using the MLF-9 lubricant mentioned in chapter 3. The combination of metal chromates with MLF-9 improved its corrosion resistance. Overcoating the lubricant with the resin-bonded Teflon material, however, provided much more corrosion protection.

The problem of corrosion encountered when using solid lubricants has been investigated by several other researchers (refs. 78 and 79). It has also been observed (ref. 80) that the use of cadmium plate in conjunction with a bonded solid lubricant reduces the wear-life of the film as much as 40 percent. The use of the above lubricant-plating combination at loads above 1000 psi is not recommended.

Mansur (ref. 81) conducted a corrosion study in which he evaluated five common lubricant films on aluminum surfaces that were exposed to humidity and salt fog. The evaluated films were (1) MoS₂ and graphite in an air-drying organic binder, (2) MoS₂ and graphite in a quick drying inorganic binder, (3) MoS₂ and graphite conforming to MIL-L-8937, (4) MoS₂ in heat-cured phenolic binder, and (5) fluorocarbon-PTFE in a heat-cured resin binder. He reports that severe corrosion occurred with the air-drying and quick air-drying MoS₂ and graphite films. Moderate corrosion occurred with the heat-cured MoS₂ and graphite films, and no corrosion was observed.
with the heat-cured MoS₂ or fluorocarbon-type films. Mansur concluded that films containing graphite should not be used on high-strength aluminum alloys.

Because bonded solid lubricants do not provide corrosion protection and may possibly cause corrosion under certain conditions, a chemical treatment or plating should be applied prior to the application of the lubricant.

It appears that thin bonded films of the lubricating plastics, such as Teflon, do protect against corrosion, mainly because they are not nearly so porous as are the MoS₂-graphite films.

From conversations with many experts in the solid-lubricant field, it seems that there has been considerable difficulty with certain non-ceramic (sodium silicate) bonded films when they are used in humid conditions or where water is present, the problem being the water solubility of the silicate material. This problem has also been encountered when attempting to test a sodium silicate bonded material at liquid nitrogen temperatures. Apparently, the problem of solubility with silicate materials can be overcome by adequate curing at higher temperatures for longer periods of time.

**EFFECTS OF OTHER THAN NORMAL ATMOSPHERE**

Solid lubricants may be required to operate not only in normal air atmosphere but in hard vacuum, inert gases, reactive gases, and radiation zones. Many of the solid lubricants will not provide lubrication in all of these atmospheres, and must be limited for use only in specific atmospheres. Graphite, for instance, does not lubricate well in a vacuum because it relies on the presence of water vapor to reduce friction.

Oxidizing atmospheres are usually detrimental to solid lubricants containing molybdenum disulfides. This is particularly true as the temperature increases, causing a rapid oxidation of the MoS₂ to MoO₃. There are solid-lubricant films, however, that work just the opposite, one being a PbO–Pb₃O₄ film developed by Peterson and Johnson (refs. 82 and 83).

The space program has generated the need for solid lubricants to be used in the vacuum of outer space. In this environment, conventional liquid lubricants are not suitable for use on moving components unless elaborate sealing procedures are followed which add unnecessary weight and complexity. Solid lubricants offer one of the most promising methods of lubrication for space systems, and at present, there are a number of research programs to develop and evaluate solid-lubricant film for use in the vacuum region of 10⁻⁶ to 10⁻¹¹ torr.
A group of materials specifically developed for vacuum operation are the MLF materials reported in chapter 3. The lubricating plastics, polytetrafluoroethylene and polyimides, are good lubricants for use in a vacuum. There are other hostile environments where solid lubricants can be considered for use, especially in the handling of reactive chemicals and gases; the use of lubricating plastics is growing in these areas.

**TEMPERATURE EFFECTS**

The resistance of various solid lubricants to extreme temperatures has significantly expedited their development. Various types of solid lubricants perform well at temperatures in the cryogenic region and at temperatures up to 1500°F. Conventional petroleum-based materials have upper performance limits of only 200°F to 300°F. Some synthetic fluids have operating ranges from 600°F to 700°F. At low temperatures, these products are limited to around −100°F, with only a few that are fluid below this temperature.

In general, the resin-bonded solid lubricants are usable over the temperature range of approximately −100°F to +450°F, but for short-term operation, they can be used at much higher temperatures. A good example of their use at extreme temperatures is as an antiseize material on threads, where very little oxygen is in contact with the material and rapid oxidation of the lubricating solid cannot take place. With the acrylic, phenolic, and epoxy resin-bonded films, it is not the lubricating solid (pigment) that is the weak part of the film but the organic binder which will deteriorate at temperatures above 450°F.

The loss of film integrity at low temperatures can be attributed to brittleness of the resin binder and at elevated temperatures to decomposition of the resin binder. At both extremes, film adhesion is very poor. The loss is much more exaggerated when the films are subjected to high loads at either low or high temperatures. The established adhesion tests described in the specifications listed in appendix A are not applicable to these conditions, since they are not conducted on parts that have been subjected to high loads at the specified temperatures.

The inorganic-bonded films (MoS₂-graphite-sodium silicate) are designed for operation over a wider extreme of temperature than are the resin-bonded materials. They are reported to be usable from −300°F to +750°F in air and to over 1200°F in vacuum (ref. 29). With these films, the lubricating solid (MoS₂) is limited in use for continuous operation above 750°F, because it rapidly oxidizes to MoO₃.
An interesting observation made by Hopkins (ref. 30) is that the friction of the sodium silicate-bonded solid lubricants decreases with increase in temperature up to about $350^\circ$ F and then begins to increase slightly. Another very desirable characteristic of these films is their compatibility with liquid oxygen, which makes them prime candidates for use on space vehicles.

The ceramic-bonded films ($\text{CaF}_2$-ceramic binder) are designed for use in the temperature range of $1000^\circ$ to $1500^\circ$ F, although they have been shown to be usable at much lower temperatures (as discussed in chapter 3). At ambient temperatures below $700^\circ$ F, these films show reduced wear-life and higher friction and, therefore, should not be considered as low-temperature lubricants. However, if the ceramic-bonded films are used at low temperatures ($700^\circ$ F) and the surface speed of the mating parts is such that high local heating is produced, the ceramic-bonded films work very well. This can probably be attributed to the melting of the binder and lubricant, forming a very thin liquid lubricant film.

All the lubricating plastics have relatively moderate temperature limits. On the high side, the addition of reinforcing materials to the plastics has not raised their useful limits much above $500^\circ$ F. On the low side, these materials have a disadvantage of increasing friction coefficient with decreasing temperature. One shortcoming of the plastic materials is their low heat-rejection rate. For this reason the surface temperatures encountered can affect the strength of these materials. Therefore, when using such materials, the designer must provide an adequate cooling system.

The powdered lubricating solids have a broad, useful temperature range. Their performance depends, to a large extent, on the environmental atmosphere as well as on the length of time at operating temperature. Molybdenum disulfide, as mentioned previously, is limited to $750^\circ$ F in an air-oxidizing atmosphere, but can be used at temperatures in excess of $1200^\circ$ F in a vacuum or when carried in nitrogen or argon gas which inhibits oxidation. Other solid lubricants show similar changes in performance depending on the environment. In general, solid-lubricant powders will perform from extremely low temperatures to extremely high temperatures, provided no chemical breakdown in their structure occurs.

**RADIATION EFFECTS**

Some radiation studies have been conducted on solid lubricants, but they have not been extensive. Consequently, the data available are fragmentary.

From the available literature, it appears that solid lubricants are
much more radiation-resistant than oils and greases (refs. 84 through 94). Goetzel (ref. 89) states that the lubricity of molybdenum disulfide is not damaged by radiation. He further reports that although Teflon is damaged to a greater extent in air than any other lubricating plastic, it is much more resistant to radiation when used in a vacuum.

Lavik (refs. 87 and 88) conducted wear-life tests on 13 commercial solid lubricants after subjecting them to radiation levels up to $10^9$ roentgens (gamma) and $3 \times 10^{16}$ nvt (neutron), and found little permanent effect on the lubricating properties of the films. The materials were of the resin- and ceramic-bonded types.

Daniel (ref. 84) conducted wear-life studies on films composed of PbO:BaO$_3$ and MoS$_2$:NaO SiO$_2$ after a gamma dose of $878 \times 10^{12}$ ergs/g (carbon) and found no perceptible change in wear-life. In additional studies, Daniel irradiated graphite in a mixed reactor flux to a neutron dosage of $3.66 \times 10^{20}$ nvt and found severe lattice damage. Molybdenum disulfide, under the same conditions, showed practically no change in lattice spacing.

McDaniel (ref. 92) conducted wear-life studies at 80°, 600° and 1200° F, using a Hohman A-6 wear tester (described in chapter 5) on five high-temperature solid lubricants after they had been subjected to a radiation dosage of $2.2 \times 10^{14}$ ergs/g (carbon) and associated neutrons of $5.2 \times 10^{14}$ N/cm$^2$. He reports that reactor radiation had no significant effect on the wear-life of lubricant films composed of PbS + MoS$_2$ + B$_2$O$_3$ or CaF$_2$ + ceramic binder. The wear-life of a film comprised of MoS$_2$ + graphite + sodium was decreased at the 80° F temperature level but improved at both the 600° and 1200° F levels. Additional radiation studies have been conducted at General Dynamics Corp. (ref. 93).

Hopkins (ref. 30) has tested lubricant MLF-5 (described in chapter 5) in an R-2 size bearing subjected to a gamma dosage of $10^{10}$ ergs/g (carbon) and $2 \times 10^{15}$ nvt fast neutrons. In comparison with the liquid lubricants evaluated at the same time, the MLF-5 gave the longest wear-life.

Pinchbeck (ref. 86) has generated radiation-effect data on several plastics, including thermosetting nylon resins, polytetrafluoroethylene, and polyethylene.

Demorest (ref. 94) has compared the wear-lives of six lubricant films that were not irradiated vs the same films irradiated at 1 MeV electron irradiation of $2.8 \times 10^{16}$ electrons/cm$^2$, and gamma irradiation of $2 \times 10^{16}$ roentgens. A statistical analysis of the wear-life data revealed that only two films were significantly affected by the radiation at the 90 percent confidence level, and that electron irradiation appeared to improve wear-life.
LOAD AND OTHER EFFECTS

The load under which solid lubricants are to operate is an important consideration. Resin-bonded lubricants can be expected to carry loads of up to 100,000 psi, while the inorganic-bonded type can operate at loads of up to 150,000 psi, as shown by Demorest (ref. 95) and Hopkins (ref. 30). In general, the operation of solid-lubricated machine elements at high loads must be at slow speed. In designing for solid-lubricant films, particularly where high loads are anticipated, the designer should always remove sharp edges that will contact the film since they will cut through the soft lubricant and plane it from the part.

Lubricating solids, such as MoS₂ in dry powder form or when used with a binder material, exhibit decreasing friction with load, as demonstrated by Hopkins (ref. 30), Demorest (ref. 95), and Karpe (ref. 96). Figure 60, chapter 9, is a graphic representation of friction vs load for typical solid-lubricant films. Speed and load effects on typical bonded solid lubricants are presented in figures 61 through 63, chapter 9.

Compared to other solid lubricants, the lubricating plastics generally perform best under light loads. Some load-carrying benefit has been achieved by the addition of fiber and metal fillers, but even then the loading cannot be excessive. The plastics are best suited for long service under light to moderate load and speed conditions.

Both the lubricating plastics and the bonded solid films should be considered for use where dirt contamination is expected because they are less affected than conventional lubricants. High-speed sliding with all the plastics and thin-film bonded lubricants must be carried out under relatively light loads.

Bonded solid lubricants have not been considered suitable for use in antifriction bearings, but some work is now being done with the silicate-bonded films that show good performance. It appears, however, that the use of lubricant composite materials as bearing retainers will be the best method of achieving dry lubrication of antifriction bearings. Vest (ref. 97) reports satisfactory results using polytetrafluoroethylene-MoS₂, fiberglass-reinforced retainers in small ball bearings. Bearings were subjected to oscillatory and slow-speed rotating motion under simulated space environment for periods of up to 12,000 hr. The bench tests conducted by Vest seem to correlate well with in-service use since many of the weather satellites used by Goddard Space Flight Center are still in operation after several years in space. Rittenhouse (ref. 90) and Clauss (ref. 98) have also reported good results using plastic and composite bearing retainers.
Novel Application Methods

One of the primary problems concerning the use of solid lubricants is the method of application to surfaces to obtain good film adhesion. In the past, adhesion of the solid-lubricant films has been obtained either by utilizing binders (e.g. thermoplastics, thermosetting resins, or ceramic binders) or by burnishing the substrate surface with the lubricating powders. There are many applications where the presence of a bonding agent is undesirable or would be a disadvantage; for example, where the performance of the film may be degraded by environmental effects, particularly those that may affect film adherence, such as decomposition of the binder. Another example where the presence of a binder is detrimental is in the coating of close tolerance parts (such as ball bearings) where the normal film thickness of 0.0002 to 0.0005 in. per surface cannot be tolerated. Burnished films leave much to be desired with respect to bonding the lubricant to the surface, and uniformity of coating.

The novel methods of application to be discussed are sputtering, in situ processing, electrophoretic deposition, and plasma spraying.

SPUTTERING

Researchers at the NASA-Lewis Research Center have conducted extensive investigations of molybdenum disulfide sputtering. Work reported in reference 99 utilized a three-electrode sputtering apparatus consisting of a thermionic cathode, an anode, and the target (fig. 49). The components of the apparatus, which are enclosed in a vacuum system, consist of:

1. Thermionic cathode—a spiraled filament of tungsten wire 0.1 cm thick, which, when heated, acts as a thermionic electron source
2. Anode—a flat stainless steel disk to which a positive potential of about 500 V is applied to establish a glow discharge between it and the thermionic cathode
3. Target—the material to be deposited, a cylindrically shaped compact of MoS$_2$ (1.3 cm in diam and about 5 cm in length), which is made without a binder by utilizing compacting pressures of 50 000 to 100 000 psi ($34.47 \times 10^3$ to $68.94 \times 10^3$ N/m$^2$)). The MoS$_2$ target is held in a water-cooled holder, in order to keep the target tempera-
ture from rising excessively. A negative potential is applied to this target.

(4) Specimen—the substrate material placed close to target material on which the atoms ejected from the target are deposited to form a continuous film.

A thermocouple gage monitors the chamber pressure during sputtering, and pure argon is admitted to the vacuum chamber through a variable leak-control valve.

Sputtering is generally performed in an argon atmosphere of several microns pressure. A potential is applied across the electrodes to ionize the gas. The cathode (target) is made of the material to be sputtered from the target, which is ejected through the plasma and deposited as a film on the specimen. The basic mechanism of sputtering is thus a process where the positive argon ions, which form a gaseous plasma, are accelerated through an electron-free region (Langmuir sheath) with sufficient energy (40 to 60 eV or $6.4 \times 10^{-18}$ to $9.6 \times 10^{-18}$ J) to knock off or sputter the negatively charged target material. In explaining the actual sputtering phenomena, present theories (refs. 100 to 102) favor a momentum exchange energy-transfer. The sputtered material is deposited on the specimen, which is placed close to the target source. For compounds, such as MoS$_2$, which have a wide disparity in vapor pressure of the constituents, the material can be deposited nearly stoichiometrically without thermal dissociation. In this method, the
basic mechanism is essentially independent of the target temperature, provided the target temperature is below the vaporization or dissociation temperature of the target material.

The bell jar is evacuated to a pressure of approximately $10^{-7}$ torr ($1.33 \times 10^{-5}$ N/m²), then purged several times with pure argon through a variable leak valve. Finally, the system is again evacuated, and argon is introduced at a controlled rate. A constant pressure of about $10^{-3}$ torr ($1.33 \times 10^{-1}$ N/m²) is maintained in the bell jar. A positive potential of about 500 V (relative to the filament) is applied to the anode (fig. 49), and the thermionic cathode emitter (tungsten filament) is heated to emission temperature causing a glow discharge. A negative potential of 2000 to 4000 V is then applied to the specimen (fig. 49), resulting in the attraction of positive argon ions from the gas discharge. The impingement of the argon ions on the surface of the specimen cleans it by sputtering. After the cleaning process has continued for about 20 min, the negative potential on the specimen is quickly switched to the target material ($\text{MoS}_2$). This procedure results in bombardment of the target by the argon ions, which are attracted by its negative potential. Molybdenum disulfide is therefore sputtered from the surface of the target and subsequently deposited on the previously cleaned specimen. The spacing between the target and the specimen is about 4 cm.

Spalvins et al (ref. 99) found that they could produce sputtered molybdenum disulfide films with thicknesses in the range of 2000 to 3000 Å. They report that the significant feature of applying $\text{MoS}_2$ in this manner is the avoidance of dissociation in the lubricant compound. X-ray analyses of the films show that nearly stoichiometric quantities of the lubricant material ($\text{MoS}_2$) were obtained.

Friction values obtained on the sputtered $\text{MoS}_2$ are from 0.06 to 0.09 which are in good agreement with values obtained on resins-bonded films, 0.03 to 0.09. There is evidence of good bonding between the sputtered films and the metal substrates as indicated by long endurance life (5 hr) of the lubricating films in friction experiments. The sputtered $\text{MoS}_2$ films have an accurate repeatability in terms of stoichiometry, adherence, thickness, and uniformity when the sputtering conditions are kept constant.

Additional work on the sputtered films has been done at the Lewis Research Center and reported in references 103 through 106. Reference 104 is of particular interest since sputtered films of molybdenum disulfide were evaluated on a variety of base metals, namely: copper, silver, beryllium, gold, rhodium, and palladium. Przybyszewski and Spalvins report (ref. 104) that the sputtered films of $\text{MoS}_2$ are not useful for the lubrication of copper surfaces. Notwithstanding, they can be successfully employed for lubricating sur-
faces of silver, beryllium, gold, rhodium, and palladium. Their data show that the sputtered films of MoS₂ were effective lubricants for the selected materials, but because of the excessive and erratic contact resistance displayed by all material combinations, the films of MoS₂ should be used only in mechanical applications and not in electric contacts. The possible exception is silver, which has an average contact resistance of 0.016 ohm. This low value of contact resistance was caused by the excessive metal-to-metal contact which occurred in this combination of metals.

Some improvements in the electrical performance of the sputtered films of MoS₂ might be obtained by sputtering silver, or some other suitable material, simultaneously with MoS₂.

Midwest Research Institute and other organizations (refs. 99 through 107) have conducted sputtering experiments. In work conducted at MRI and reported in reference 107, Falex test specimens were coated with sputtered MoS₂ films and evaluated under standard test conditions at a 1000-lb jaw load in accordance with reference 137C. Wear-lives of over 90 min were obtained, the magnitude of which compares quite favorably with those obtained on many resin-bonded films conforming to such specifications as MIL-L-8937 and MIL-L-46010, described in appendix A. The work conducted at MRI also indicates that it is possible to sputter other solid lubricants, namely, calcium fluoride.

It appears that the physical dc sputtering method of applying lubricating solids to metal substrates is practical, particularly in areas where only extremely thin films can be tolerated such as close-tolerance bearings (antifriction, ball, and roller). To date, however, very little actual evaluation has been done on sputtered films in bearing applications. One reason for this lack of test data is the fact that films could not be sputtered on curved or irregular surfaces without elaborate means of turning the specimens inside the vacuum system during the sputtering process. During a visit to the Lewis Research Center, the author was informed by those engaged in the sputtering program that they have devised a method of depositing the lubricant material evenly on all surfaces of the part being coated. If they, in fact, are able to coat evenly in all directions, this method certainly will be a major step in the use of the sputtered films.

**IN SITU PROCESS**

A solid-lubricant film can be formed on metal surfaces from the surrounding atmosphere through the use of reactive or film-forming gases. Vest (ref. 108) has discussed a method of depositing an MoS₂ lubricating film by first electrodepositing a coating of MoO₃, and
then subjecting the coated part to H$_2$S gas at elevated temperature and pressure. The process results in the formation of a complex molybdenum disulfide crystalline structure that incorporates the metal substrate at the treated surface. Film thickness can be controlled accurately by this process; for instance, a coating thickness of from 0.00005 to 0.0005 in. can be successfully applied uniformly. A commercial process identified as E3C* by its processor is available. Vest and the processor (ref. 109) both report that the material can be applied to stainless steels 303, 304, 316, 416, and 440C; aluminum alloys 2024, 6061, and 7075; and to both mild steel and M-10 tool steel. The processor (ref. 109) further states that all conductive metals can be successfully processed without difficulty except copper and its alloys, which do not coat satisfactorily. Vest further reports that the coefficient of friction of the in situ film is 0.025 to 0.05 and that film wear is much better than the inorganic-bonded molybdenum disulfide films. He has also successfully applied the in situ films to instrument bearing races, retainers, and small gears.

The processor of the E3C process gives (ref. 109) several design factors that must be followed when considering the process for use on bearing parts: (1) the base material must withstand temperatures of 350° F for up to 3 hr without damage, (2) covered or concealed areas cannot be properly coated, (3) the process makes it difficult to mask portions of the surface where coating is not desired, and (4) finely ground or polished surfaces require special pretreatment. When properly applied, the material has excellent film adhesion and gives test wear-lives equal to or better than conventional baked-on bonded solid lubricants.

**ELECTROPHORETIC DEPOSITION**

Coating by electrophoresis, in which small suspended particles are deposited on an electrode with the aid of an electrostatic field, is well known. The process is most widely used in biological applications as an accurate analytical tool to separate the components of solutions. Electrophoretic deposition has been used for many years by the radio-tube industry in coating delicate tube parts and by the rubber industry in producing rubber gloves.

A solid lubricant, such as MoS$_2$, can also be applied by electrophoresis. The lubricant can be applied by itself or with a binding agent. The carrier solvent used in the process should be of the organic type with a high dielectric value, such as acetone and alcohol.

* E3C Process—Proprietary process of Dow Corning Corp.
The lubricating solid is placed in the solvent and agitated, because a good suspension is imperative for the process to work. There are a number of ways that the quality of the suspension can be improved, among which are (1) the use of a finely divided lubricating material (0.5 μ or less) and (2) the addition of a protein material (zein) which will help form a colloidal suspension. When a good suspension is obtained, the part to be coated is placed in the solution and used as one of the electrodes. A second electrode is connected and a high-voltage charge is passed through the solution for a controlled period of time, causing the lubricant particles to migrate through the solution and coat the test part. Parts are then removed from the solution and dried or fired prior to use. A schematic illustration of a typical electrophoretic setup is shown in figure 50.

The electrophoretic process has been employed by a number of investigators (refs. 110 through 114). The Vitro Laboratories of the Vitro Corp. of America have done considerable research in this field and have obtained a number of U.S. patents (refs. 115 and 116) on the process. In general, MoS₂ is co-deposited with a metal oxide. The metal oxide is then reduced in a hydrogen furnace and the metal becomes the binder for the lubricant (MoS₂) in the coating.

The advantages of this method of coating include: good reproducibility of coating thickness, rapid coating application, and good coating uniformity. Of these advantages, coating uniformity is especially significant with complex shapes such as gears. Coating uniformity is obtained by means of the electric insulating properties of the applied coat. As some of the particles migrate to the attracting electrode and deposit, they partially insulate that portion of the surface. The particles then approaching the electrode move to the uninsulated portion, thus building up a uniform coating.

Figure 50.—Setup for applying solid-lubricant films electrophoretically.
McConnell et al (ref. 117) report the use of the electrophoretic process to overcoat a ceramic-bonded solid lubricant with a film of molybdenum disulfide. Their intent was to fill the voids of the porous ceramic-bonded films with the electrophoretically deposited film and thus increase the film's useful wear-life. They report a substantial increase in the wear-life of their original ceramic film and state that no increase in film thickness was found. It therefore appears that they accomplished the original intent of the overcoating, that is, to fill the void areas with extra lubricant.

Although the application of solid lubricants by the electrophoretic process has shown some promise, it has not yet become a commercial reality.

PLASMA-SPRAY PROCESS

Although the techniques of plasma and flame spraying are not new, their use in applying solid lubricants has not been thoroughly explored. The plasma-spray process, a unique method for the application of a wide variety of solid-film lubricants, has several advantages over conventional ones. Plasma-applied films require neither organic solvents nor water suspensions and, therefore, require no bakeout. Probably the most important feature of the flame-spraying process is the ability to apply such coatings as ceramics to metal substrates without affecting the hardness of the substrate material. Normally, ceramic materials are prepared from frits and applied on a surface by firing at extremely high temperatures. These temperatures are often above the heat-treatment temperature of the base material, and thus may affect its mechanical properties. As stated above, the flame-spraying process eliminates this problem. Lubricants that have been applied by this process range from plastic to metal and ceramic binders combined with solid lubricants, such as molybdenum and tungsten disulfides and graphite.

Two processes appear useful for applying solid lubricants by flame-spray or plasma-stream techniques: (1) application of a porous ceramic coating followed by burnishing a solid lubricant onto the surface and into the porous structure; and (2) application of a mixture of a binder and lubricant powder simultaneously. The latter process eliminates the curing step. The second process, however, has some shortcomings, particularly that some of the lubricant powder is altered by the heat required for spraying, resulting in films that are overrich in binder; either ceramic or metal binders can be used.

Hopkins et al (ref. 118) conducted a feasibility study of applying solid lubricant films with a plasma gun. The gun configurations used to apply metals, resins, ceramics, and solid lubricant (graphite,
WS₂, and MoS₂) films are described. The importance of the spray-gun configuration in avoiding lubricant degradation and/or dissociation as well as in obtaining good binder fusion is delineated. Their findings were: (1) a dual-port entry gun, which introduced binders in the hot zone and lubricant powders in a cool zone, was the best for the application of ceramic-bonded MoS₂ films; and (2) the film adhesion, load-carrying capacity, wear-life, and other properties of plasma-sprayed lubricants were not as good as those of lubricants applied by conventional methods.

Kremith et al (ref. 119) also experimented with plasma-sprayed solid-lubricant films. Kremith used as his lubricant powders composite materials of copper, nickel, and silver with molybdenum disulfide. In his opinion, a composite powder provided some protection for the lubricant phase and it also decreased material segregation during the spray operation. Hopkins (ref. 118) mentions that at least one organization in the U.S. now uses a plasma-spray process in applying conventional bonded solid lubricants.

It has been demonstrated (refs. 118 and 119) that the possibility of lubricant degradation or dissociation does exist when using the plasma spray to apply the conventional lubricating solids such as MoS₂, WS₂, and graphite. Therefore, it is the author’s opinion that the higher-temperature lubricant powders such as CaF₂, SrF₂, and BaF₂ should be used in the plasma-spray process rather than MoS₂ and WS₂.
Present Applications for Solid Lubricants

Knowledge of the use of solid lubricants by governmental agencies (NASA and DOD*), aerospace and commercial industries, as well as information obtained from literature searches indicates that the use of all forms of solid lubricants is increasing. The general acceptance of solid lubricants has been rather slow, but within the last several years their potential has been more fully realized.

This chapter discusses a few areas in which solid lubricants have been used successfully. It is hoped that the reader who is not familiar with the subject will be able to relate some of the examples to his own particular lubrication problems.

**AEROSPACE AND MILITARY APPLICATIONS**

All types of solid lubricants are used in the aircraft industry on both military and commercial aircraft, as well as in missiles and space vehicles. The use of solid lubricants in this industry has been expedited by the need for lubrication under extreme environmental conditions.

The use of solid lubricants for military aircraft has increased steadily over the years from only a few applications on the early 1900's fighter planes to over 1000 applications on the North American Rockwell XB-70. Approximately 95 percent of the plain bearings on the XB-70 were either of the lubricating-plastic or thin-film-ceramic solid-lubricant variety. The North American Rockwell Corp. has made extensive use of its own developed ceramic-bonded lubricant material, "Vitrolube," which has been shown to be a good lubricant from room temperature up to 700° F. The uses of this material are generally on bearings of the plain bushing or plain spherical type. Many aircraft applications are on such safety devices as ejection seat parts, canopy linkage, and control lever components, where movement is limited, but where the mechanism must work when needed.

Many reports exist describing applications of solid lubricants in missiles, satellites, and spacecraft. A large number of these appli-

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* DOD—Department of Defense.
cations are of the plastic-bearing variety. Goetzal et al (ref. 89) and
Clauss (ref. 98) report that a wide variety of materials have been
evaluated at the Lockheed Missiles and Space Co. (now Lockheed
Aircraft Corp.) for use in space applications.

Vest and Evans at the Goddard Space Flight Center indicate that
nearly all of their work in the lubrication of unmanned satellites
is directed toward the use of solid lubricants. They have been pri-
marily interested in lubricating small ball bearings and have been
evaluating plastic materials. It is interesting to note that several
of the satellites under the jurisdiction of the Goddard Space Flight
Center have been in orbit for many years and that simulated testing
of bearings has correlated well with actual use in satellites.

Midwest Research Institute has developed several lubricant coat-
ings that are used in launch and space vehicles. Typical applica-
tions are: gimbal bearings, tie-down clamps, camera parts, gears
and bearings, sliding tracks, and valves.

The U.S. Army also uses large quantities of solid lubricants in
various forms on weapons and ground-handling equipment. Meade
and Murphy (ref. 76) list over 40 such applications, including:

| Ball sockets        | Trailers—         |
| Sliding covers      | elevating mechanisms |
| Handwheels          | torsion bar suspensions |
| Bearing-support points | bearing areas    |
| Slide dust covers   | Rifle mounts      |
| Hand brakes         | Rocket launchers  |
| Chain and sprocket drives | Ammunition   |
| Plungers            | Firing mechanisms |
| Screw threads       | Breech mechanisms |
| Telescoping-tube assemblies | Trunnion bearings |
| Warhead-mating fixtures |

Some work is also being done by the Army on the lubrication of
helicopter gear boxes with solids in the form of composite gears
and composites as cage materials in antifriction bearings. The ap-
plication in helicopters has not been to replace the oil-lubricated
system now in use but as a backup system that will allow the aircraft
to return to earth if fluid lubrication is lost.

The U.S. Navy is also a large user of solid lubricants for aircraft
and ships. Personnel of the Naval Air Engineering Center state
that the Navy uses in excess of 100 000 lb/year of greases contain-
ing molybdenum disulfide. This Center is a leader in the develop-
ment, evaluation, and usage of the solid-lubricant materials. The
Center also has the responsibility for maintaining several of the
military specifications covering solid-lubricant films.

The consensus of the military agencies and aerospace industries
contacted during this survey indicates that use of these materials is increasing and will continue to increase.

**COMMERCIAL APPLICATIONS**

There is a very large market in the commercial field for solid lubricants, including such industries as automotive, heavy electrical, metal-working, marine, household-appliance, toy, farm-machinery, and solid-waste disposal. It is not the intent of the author to include every application, but only to alert readers to the possibility of using solid lubricants in their own work. The areas where these lubricants have been found useful are given below, and there are undoubtedly many more.

- Convex draw dies
- Solenoids
- Arc-furnace guides
- Core-oven wheel bearings
- Conveyor wheels
- Railroad centerplates
- Helicopter gear boxes
- Cold extruder (wire)
- Roller chains
- Turntables, 45-rpm spindle (music)
- Air-cooled, 4-cylinder engines
- Cold hobbing (dies in steel)
- Rolling mills
- Conveyor parts
- Brake air cylinders
- Oil pipeline valves
- Bullets
- Boat hulls
- Roll-extruded cylinders
- Wire rope (wire strand)
- Aircraft-landing gears
- Oxygen-furnace bearings
- Tee connectors, gas main under pressure
- Small arms
- Ball joints and front suspensions (autos)
- Air cylinder tubing
- Three-jaw chucks
- Plug valves
- Race car axle threads
- Timing mechanisms
Fuel-pump mechanisms
Rear-axle assemblies (tractors)
Electrical connectors (space)
Cylinder walls, gasoline engines
Gear trains
Clutch brakes
Hinge bolts, pylon-hinge pins (copters)
Rocket bellows
Bearing-link assemblies
Clock springs
Camera shutters (space flights)
Root ribs, aircraft
Rocket engines, fasteners, valves, and bushings
Rolling-mill couplings
Self-aligning ball bushings
Composite brushes
Thrust bearings
Draw and blanking punches
Drilling and tapping machines
Wheel bearings and king pins (truck fleets)
Sliding-tandem trailers (trucks).
Designing for the Use of Solid Lubricants

This chapter includes a discussion on how bonded solid lubricants can be used effectively. Guidelines that can help the user obtain the best results with thin-film bonded solid lubricants are also discussed. Performance, especially durability, of these solid-lubricant films, is affected by (1) compatibility, (2) substrate hardness and pretreatment, (3) bearing clearance and film thickness, (4) wear debris, and (5) effects of load, speed, and temperature.

COMPATIBILITY CONSIDERATIONS

When bonded solid-lubricant films are used, their compatibility with the environment must be considered, for example, the use in the presence of liquid oxygen (LOX). Compatibility with LOX is of such importance that all materials used which might come into contact with it must first pass the compatibility test explained in reference 120. The normal resin-bonded (phenolic, epoxy, and polyimide) solid-lubricant films are not LOX-compatible and should never be used in liquid oxygen or high concentrations of gaseous oxygen because the slightest impact can cause an explosion. Only the inorganic- and ceramic-bonded films have been shown to be LOX-compatible. If there is a question of a material being compatible with liquid oxygen, the user should refer to reference 121.

The use of ethylene oxide to sterilize for medicinal purposes when it is not feasible to heat-sterilize is another example in which compatibility with the environment must be considered. Constituents in bonded solid-lubricant films such as antimony trioxide may react chemically with ethylene oxide to cause deterioration in the structural properties of the film which, in turn, shortens the wear-life of the film.

Oils and greases and, in fact, most liquids are very harmful to most bonded solid-lubricant films, and extreme care should be taken to prevent the contamination of these films under operating conditions. Tables XIII and XIV contain data obtained at Midwest Research Institute showing how oil and grease contamination drastically reduces load-carrying capacity and wear-life. This liquid contamination can affect the bonded lubricant films in three ways:
### Table XIII.—Falex Load-Carrying Capacity Tests

<table>
<thead>
<tr>
<th>Lubricant designation</th>
<th>Load-carrying capacity in lb. Jaw load in air</th>
<th>Load-carrying capacity in lb. Jaw load in SAE 30 wt oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air-cured film conforming to MIL-L-23389</td>
<td>1500</td>
<td>1250</td>
</tr>
<tr>
<td>Heat-cured phenolic bonded film conforming to MIL-L-8937</td>
<td>2600</td>
<td>1600</td>
</tr>
<tr>
<td>Heat-cured phenolic bonded film conforming to MIL-L-8937</td>
<td>3000</td>
<td>1625</td>
</tr>
<tr>
<td>Sodium silicate binder</td>
<td>1500</td>
<td>1125</td>
</tr>
<tr>
<td>High-temperature polyimide binder</td>
<td>4500</td>
<td>2950</td>
</tr>
</tbody>
</table>

Notes:

1. All loads are averages of at least six tests.

### Table XIV.—Falex Wear-Life Tests

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Phenolic bonded film</td>
<td>110</td>
<td>3.0</td>
</tr>
<tr>
<td>Phenolic bonded film</td>
<td>248</td>
<td>3.0</td>
</tr>
<tr>
<td>Sodium silicate bonded film</td>
<td>10</td>
<td>2.5</td>
</tr>
<tr>
<td>Inorganic air-dried film (proprietary)</td>
<td>406</td>
<td>11.0</td>
</tr>
<tr>
<td>Polyimide bonded film</td>
<td>502</td>
<td>4.5</td>
</tr>
<tr>
<td>Sputtered MoS₂ film (no binder)</td>
<td>90</td>
<td>3.0</td>
</tr>
</tbody>
</table>

Notes:

1. All times are averages of at least six runs.

(1) it may introduce hydraulic forces which tend to break up the film as it passes through a loaded zone, (2) it may soften the film and thus weaken it structurally, and (3) it may increase the friction between the bearing surfaces. If the bonded films are inadvertently contaminated with oils or greases, they can be cleaned with a suitable volatile solvent and restored to a usable condition with little loss of wear properties. Contamination in the form of grit should also
be avoided when using bonded lubricant films because hard particles will become imbedded in the surface and scratch the relatively soft film.

**EFFECTS OF SUBSTRATE AND MATING-SURFACE CONDITIONS**

Machine designers or users of bonded lubricant films should understand that the bond between the substrate surface and the lubricant film is primarily one of mechanical adherence. Therefore, surfaces to be coated with a bonded lubricant film must be pretreated prior to receiving the film. Pretreatments usually involve roughening of the surface by either wet- or dry-grit blasting. Sometimes chemical treatments, such as phosphating or anodizing, are used. In general, pretreatment increases the interfacial surface area and enhances interlocking between the lubricant film and substrate.

These pretreatments will effectively improve the durability of bonded solid-lubricant films, but there is frequently a wide variation in the quality of these pretreatments, particularly the chemical ones. Wet- or dry-grit blasting produces the most uniform and reproducible pretreatment of substrates for solid lubricants. Blast pressure, blast distance, and quality (sharpness and hardness) of blast media are easily controlled. Chemical treatments are done in a liquid bath which requires constant scrutiny to insure the proper chemical mix, reaction speeds of which can vary from one substrate material to the next. The chemical treatments must also be compatible with the substrate material with which they are used, for example, some anodizing processes will weaken certain aluminum alloys.

Chemical pretreatments and platings are generally used on substrates for bonded solid-lubricant films if there is a requirement for corrosion protection, because the bonded lubricants do not provide protection from corrosion when surfaces to be coated are not pretreated.

At times the user must consider a pretreatment other than those most favorable for long film durability; consequently, it is desirable to know the effect these pretreatments have on film wear-life so that the best compromise with the imposed restrictions can be selected.

In general, bonded solid-lubricant films applied to hard surfaces last longer than those applied to soft surfaces. This fact has been demonstrated in laboratory tests for both heavy and light loads (refs. 16 and 18), as well as in service. Effects of substrate hardness have been investigated under light-load conditions at Midwest Research Institute (ref. 23). In many applications only one of the two surfaces in a bearing is coated; therefore, the effect of hardness and
roughness of the uncoated surface on the wear-life of a solid-lubricant film needs to be known. Such an investigation was conducted by Gaddis and Hopkins (ref. 18). In this study, a solid-lubricant film (MLF-5) for the Marshall Space Flight Center (ref. 23) was applied to the end of pellets (1⁄4-in. diam steel pins) that were slid over a wear plate of selected hardness and roughness. Two levels of substrate and mating-surface hardness and several levels of mating-surface finish were used. Sufficient tests were run to permit the data to be analyzed statistically, and the results are shown graphically in figure 51.

The combination of hardened pellets on hardened wear plates appears to be the best selection of a substrate and mating-surface combination under the conditions of the MRI investigation. Hardened pellets coated with MLF-5 and rubbed against a hardened wear plate having a surface finish of 2 to 4 \( \mu \) in. rms provided the longest wear-life. Longer average wear-lives were obtained with hardened wear plates than with annealed wear plates. With the exception of hardened pellets on annealed wear plates, the recommended surface finish for the longest wear-life is between 2 and 4 \( \mu \) in. rms. Considering all hardness combinations, it appears that the mating-surface roughness should not exceed 5 \( \mu \) in. rms or be less than 1 \( \mu \) in. rms.

The work just described is for one particular solid-lubricant film, and the conditions (substrate hardness, mating-surface hardness, and mating-surface finish) found to be the best for long wear-life may not be the same for other films. In addition, the effect of varying the substrate and mating-surface material, test load, test temperature, sliding speed, and contact configuration were not determined. With the lack of more definitive information, however, these results constitute the best guide for selecting substrate hardness and mating-surface hardness and finish.

**BEARING CLEARANCE AND FILM THICKNESS**

The thickness of bonded solid-film lubricants is much greater than a lubricating film of oil or grease. Consequently, sufficient clearance must be allowed in the bearing design to accommodate the bonded film. The recommended thickness for most solid lubricant films is about 0.0005 in. per surface. The thickness of lubricant material that will provide the most durable film, however, will vary from film to film and from application to application.

Numerous investigators have studied the effect of film thickness on the wear-lives of bonded solid lubricants, often reporting contradictory results. Recently, Jones (ref. 122) found that wear-life was directly proportional to the square root of the thickness of the lubri-
Figure 51.—Comparison of effects of substrate hardness, mating-surface hardness, and surface finish on the wear-life of MLF-5 lubricant.
cant coating. His work was conducted on a phenolic bonded film using a modified LFW-1 test machine in an oscillatory configuration. Rabinowicz (ref. 123) investigated the effect of film thickness using a pin-on-disk machine and reported that total wear-life increased with film thicknesses. Hopkins (ref. 30) has reported similar results with a three-pellet apparatus running under light loads.

The author has conducted an extensive investigation of the film-thickness effect using three different test configurations (figs. 52, 53, and 54), widely used in solid-lubricant film research and development. The lubricant used in this evaluation was a polyimide-resin
bonded material composed of molybdenum disulfide and antimony trioxide (MLR-2). The lubricant was developed at Midwest Research Institute for the Marshall Space Flight Center. This particular film has good homogeneity and shows good repeatability in wear-life tests. Substantial differences in wear-life vs film thickness resulted from the three test configurations. The high-load configuration in figure 53 showed the longest wear-life at film thickness between 0.0002 and 0.0003 in. per surface. The intermediate load configuration, figure 52, showed the longest wear-life at film thickness between 0.0003 and 0.0004 in., whereas the lightly loaded configuration, figure 54, indicated an increase in film life with increasing film thickness. Results of these tests are shown graphically in figures 55, 56, and 57. It is uneconomical to apply film much thicker than 0.001 in. per surface. In addition, the designer-user must remember that a thicker film will result in a substantially larger amount of troublesome wear debris.

Wear-life of the bonded solid-film lubricants is generally extended by coating both parts in any mating combination. A statistically designed program has been conducted at MRI to determine which parts in a bearing combination should be lubricated. In this unpub-

![Graph](image)

**Figure 55.**—Effect of film thickness on Hohman wear-life of MLR-2 lubricant. Hohman A-3 dual rub shoe. Test conditions: 300-lb load, 220 rpm (79.2 ft/min), at room temperature. Test specimens: ring 440CRc 55–60; rub shoes, 440C Rc 55–60.
lished work, two test configurations were selected: a journal bearing and a flat sliding on a flat. The surfaces considered for lubricant coating were (1) all mating surfaces; (2) the rotating or sliding member only, and (3) the stationary member only. In addition, the effects of film thickness were also investigated at thicknesses of 0.0002 to 0.001 in. per surface. In both test configurations, the longest life was achieved with all mating surfaces coated; the next longest life was obtained by coating only the moving surface. Coating only the stationary member resulted in the shortest life, indicating that the surface or surfaces that present the greatest volume of film to the rubbing contact have the longest life. The best results were obtained with a coating thickness of 0.0002 in. (all surfaces) per surface on the flat-on-flat configuration while a coating thickness of 0.0005 in. (all surfaces) per surface was best on the journal-bearing configuration.

Although the film thicknesses discussed above might appear difficult to obtain, an experienced spray operator with proper equipment can apply films to tolerances of \( \pm 0.0001 \) in.
WEAR-DEBRIS EFFECTS

In designing for the use of bonded solid lubricants, it must be kept in mind that the bonded lubricants are soft, relatively weak materials. As they are worn away during use, the wear products, in most cases, should be removed from the mechanism. The texture and porosity of the lubricant films will vary from one to another. Some materials appear fluffy when viewed under a microscope while others look quite dense. When subjected to a load, cured solid-lubricant films can compact from 10 to 30 percent. The fluffy looking films experience the greatest compacting and produce the most wear debris during initial use. This debris usually increases the rate at which additional wear debris is generated. The effect of wear debris on film durability is most severe when the film contains hard particles of metals, oxides, silicates, ceramics, or the like. Such particles often come from the binder which accounts for about 20 percent of the volume of the film.

Wear debris is generated by the sliding action of a sharp edge in
contact with the surface of a bonded film on a bearing surface. Such sharp edges result in a cutting action, shearing the film from the substrate. A rolling contact over a sharp edge on a coated surface tends to chip the film from the surface and, in many cases, starts a failure point. Film contact with sharp edges should always be avoided. This can usually be done simply by including a note on drawings to break all sharp edges. The generation of normal wear debris in an oscillatory-sliding operation is shown in figure 58.

Some provisions must be made for the removal of wear debris during both the initial run-in period of the film and during the entire usable life of the lubricant. Run-in wear debris can often be blown or brushed out of an assembly after the initial operation. Methods of handling the wear debris need to be considered during the design of the parts to be coated. In some instances, cavities could be added in the surface to collect debris. The benefits of such cavities have been substantiated by tests conducted at MRI and by discussions with investigators in other laboratories. In assemblies, it may be necessary to provide shielding of certain machine elements from the wear debris generated in other parts, particularly if some moving elements are located below and in line with the areas where wear-debris generation is expected. Past experience has shown that wear particles generated from lubricant films have caused bearings to jam in certain operations.

![Figure 58. Generation of wear debris.](image-url)
Wear debris may not always be detrimental as has been demonstrated in unreported journal-bearing work at MRI. The test was stopped periodically and all wear particles were removed from the bearing area. In such cases, bearing life was substantially reduced from that obtained when the wear particles were allowed to remain. Therefore, it seems advisable to use a bearing with a center groove to collect the wear particles with the expectation that they will gradually work back into the load area and contribute to the useful life of the film.

![Graph](image-url)

**Figure 59.**—Effect of temperature on wear-life of typical bonded lubricant films.
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TEMPERATURE, LOAD, AND SPEED CONSIDERATIONS

Temperature, load, and speed will generally affect the wear-life of bonded solid-film lubricants. In addition, these factors can be expected to interact (combined and related effects). Of these, temperature probably has the most pronounced effect; this effect on the wear-life for four films is shown in figure 59. As depicted in the figure, there is a drastic decrease in wear-life in air with increased temperature. A similar effect, perhaps even greater, can be expected for most available solid-lubricant films. The reduction in wear-life caused by elevated temperature is thought to be primarily the result of two factors: (1) the binders used in the films lose bond strength with increased temperature and (2) the lubricating pigments (MoS₂ and graphite) tend to oxidize more rapidly at elevated temperatures.

Because temperature is known to have a large effect on wear-life, the operating temperature should be kept as low as possible. Bearings lubricated with bonded solid-film lubricants tend to operate at higher temperatures than comparable oil- or grease-lubricated

<table>
<thead>
<tr>
<th>Lubricant film identification</th>
<th>Temperature, °F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Room temperature</td>
</tr>
<tr>
<td>MoS₂-graphite in air-drying binder (MIL-L-23398)</td>
<td>0.05</td>
</tr>
<tr>
<td>MoS₂-graphite, sodium silicate binder</td>
<td>0.08</td>
</tr>
<tr>
<td>MoS₂-graphite, phenolic (MIL-L-8937)</td>
<td>0.07</td>
</tr>
<tr>
<td>MoS₂-graphite, gold, sodium silicate binder MLF-5*</td>
<td>0.08</td>
</tr>
<tr>
<td>MoS₂-Sb₂O₃, high-temperature polyimide binder (MLR-2)*</td>
<td>0.04</td>
</tr>
</tbody>
</table>

Notes:
(1) Tests conducted on double rub-shoe test machine (Mark V) described in chapter 6.
(2) Test conditions: 100-lb load at 500 rpm.
(3) Three repeat tests over entire range.
* Films developed by Midwest Research Institute for Marshall Space Flight Center.
bearings because the bonded films usually exhibit higher friction and are poor thermal conductors. In the case of liquid-lubricated systems, the liquid lubricants are often used to cool the bearings; because of their inherent nature, bonded solid-film lubricants will not serve this purpose. For this reason, the bearing location and housing should be designed to take advantage of other methods of heat rejection. For example, the bearing housing might be made with a large cross section and have large external surface area. Ventilation should be provided for the bearing by using radiator fins or a cooling fan. The effect of temperature on the coefficient of friction of a typical bonded solid-film lubricant is shown in table XV. The effect of pressure on the coefficient of friction of a typical bonded solid-film lubricant is shown in figure 60.

In general, the wear-life of bonded solid-film lubricants is decreased as the load and/or speed is increased. The load and speed effects on a phenolic bonded film conforming to MIL–L–8937 are shown in figures 61 and 62. The same effects are shown in figures 63 and 64 on a high-temperature polymeric bonded film developed by MRI for NASA (ref. 20). Data for the above-mentioned figures were generated on a test machine using a \( \frac{3}{8} \)-in. journal-bearing
arrangement where both shaft and inner diameter of the bushing were coated with the lubricant material. The wear-life of the film conforming to the MIL-L-8937 specification was substantially reduced by increases in both load and speed. The other two materials show improved wear-life with increased load and both show decreases in wear-life with increased speed.
Figure 63.—Speed vs wear-life of a polymeric bonded film.

Figure 64.—Load vs wear-life of a polymeric bonded film.
Specifications

The widespread use of dry, solid-lubricant powders, thin-film bonded lubricant coatings, and reinforced plastic materials, discussed in the preceding chapter, has resulted in a number of military and industrial specifications covering their methods of application and use.

The specifications include, but are not necessarily limited to, the ones discussed in this chapter. It is not practical to reproduce the specifications in their entirety for this survey—only highlights of the requirements will be given. Materials covered include: lubricant powders, thin films (organic- and inorganic-bonded), plastics, and solid lubricants as additives to greases and oils. The reader can obtain copies of the specifications from the agencies listed in table XVI.

A word of caution is offered in regard to the use of the following specifications. Neither the specifications nor the materials qualified to them are cure-alls for every application. Nevertheless, when used in a wise and judicious manner, they can be very helpful in the selection of specific solid lubricants for particular uses.

SPECIFICATION BRIEFS

**MIL-M-7866: Molybdenum Disulfide, Technical, Lubrication Grade**

This specification covers the requirements for procurement of one grade of powdered molybdenum disulfide to be used for the lubrication of surfaces when boundary conditions exist. The powder shall have a purity (98.5 percent pure MoS₂, minimum) and a particle size (average, > 5 µ and 10 µ) suitable for general lubricating use.

*Uses.*—MIL-M-7866 is intended for use as a dry lubricant or as a component with suitable specification oils or greases for special applications where other lubricants are not satisfactory. It reduces friction and wear under low- and high-sliding velocities; is used as thread antiseize for lightly loaded applications where fluid lubricant is objectionable; and is an effective lubricant over a wide range of temperatures.

*Limitations.*—The unbonded lubricant does not give corrosion protection. The material must be bonded and cured to develop
### Table XVI.—Specifications

<table>
<thead>
<tr>
<th>Specification no. and title</th>
<th>Ref. no.</th>
<th>US Government agency</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIL—L—25504 (USAF): lubricant, solid film, heat-cured [superseded by MIL—L—8937 (ASG)]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MIL—L—46009 (MR): lubricant, solid film, air-drying (in pressurized containers) [superseded by MIL—L—23398]</td>
<td></td>
<td></td>
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<tr>
<td>MIL—L—81239 (WEP): lubricant, solid film, extreme environments</td>
<td>130</td>
<td>NASA—George C. Marshall Space Flight Center, Huntsville, Ala. 35812</td>
</tr>
<tr>
<td>MSFC—SPEC—253A: lubricant, dry film, ceramic, MLF—5 and MLF—9 (preparation and application)</td>
<td>131</td>
<td>NASA—George C. Marshall Space Flight Center, Huntsville, Ala. 35812</td>
</tr>
<tr>
<td>MSFC—SPEC—502: lubricant, dry film, ceramic, MLF—5 (preparation and application—proposed)</td>
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**Table XVI.—(Concluded)**

<table>
<thead>
<tr>
<th>Specification no. and title</th>
<th>Ref. no.</th>
<th>US Government agency</th>
</tr>
</thead>
<tbody>
<tr>
<td>MSFC-SPEC-81205: bonded solid dry-film lubricants, material specification for and application of bonded solid dry-film lubricants</td>
<td>133</td>
<td>NASA-George C. Marshall Space Flight Center, Huntsville, Ala. 35812</td>
</tr>
<tr>
<td>MIL-G-21164: grease, molybdenum disulfide (for low and high temperatures)</td>
<td>134</td>
<td>Bu Weps Engineering Standardization Office (Code X) Naval Air Engineering Center, Philadelphia, Pa. 19112</td>
</tr>
</tbody>
</table>

Maximum lubrication potential; in this form it must also provide a minimum of 500 hr of corrosion protection under conditions of heat and high humidity. Mixtures of this powder with oils or greases in field applications where performance data have not been established is not recommended.

**MIL-G-6711: Graphite, Lubrication**

This material is a 200-mesh, high grade, powdered graphite for use preferably as a dry lubricant, but may be mixed with a proven specification oil.

**Uses.**—The intended use of MIL-G-6711 is principally as a "dust-on" or "brush-on" dry-powder lubricant for sliding surfaces (i.e., tracks, slides, and grooves). It may be used over a wide range of temperatures.

**Limitations.**—This material is a large particle size powder and is not recommended for blending with oils or greases, particularly, in aircraft or aerospace applications. MIL-M-7866 molybdenum disulfide should be used in preference to this material, if possible.

**MIL-L-8937 (ASG): Lubricant, Solid Film, Heat-Cured**

This specification establishes the requirements for a solid film lubricant intended to reduce wear and prevent galling and seizure of metals. Condensed specification requirements, uses, and limitations are given below.
Film appearance and thickness.—The bonded film lubricant shall appear smooth and free from cracks, scratches, pinholes, blisters, bubbles, runs, sags, foreign matter, grit, rough particles, separation of ingredients, and other imperfections.

Film adhesion.—The bonded solid-film lubricant shall not be lifted from the test panel by the pressure-sensitive masking tape method. A uniform deposit of powdery material may cling to the tape, but lifting of any flakes or particles which expose any bare metal shall indicate unsatisfactory adhesion. Test should be made in accordance with reference 137A.

Thermal stability.—The bonded solid-film lubricant shall not flake, crack, or soften; and shall have satisfactory adhesion when tested for 3 hr at −65°F, in accordance with reference 137A.

Fluid resistance.—The bonded solid-film lubricant shall not soften, lift, blister, crack, or peel; and shall have satisfactory adhesion when half immersed for 24 hr at room temperature in each of the following fluids—standard hydrocarbon test fluid, aviation gasoline, jet fuel, hydraulic fluids (petroleum and nonpetroleum base), aircraft lubricating oils (petroleum and synthetic base), silicone fluid, and trichloroethylene.

Endurance life.—The bonded solid-film lubricant when tested on the Falex Lubricant Tester shall have an average life of not less than 60 min at 1000-lb gage load, in accordance with reference 137C. The minimum life of any single run shall not be less than 50 min.

Load-carrying capacity.—The bonded solid-film lubricant when tested on the Falex Lubricant Tester, in accordance with reference 137D, shall have a minimum load carrying capacity of 2500-lb gage load.

Corrosion resistance.—The bonded solid-film lubricant on anodized aluminum panels shall show or cause no discoloration, pitting, formation of white deposits or other evidence of corrosion after 500 hr at 120°F and 95 percent humidity, when tested in accordance with reference 137E.

Uses.—This solid-film lubricant is intended for use on steel, titanium, aluminum, aluminum alloys, and other metals. It is useful where other lubricants are difficult to apply or where they may be contaminated by dirt and dust. The lubricant is suitable for sliding motion surfaces, such as plain spherical bearings, flap tracks, hinges, and cams.

Limitations.—This solid-film lubricant should not be used with oil or grease unless experience indicates otherwise. Because of the 300°F cure temperature, it should not be used on materials which are adversely affected by exposure to this temperature. It should not be used where there is potential contact with liquid oxygen. The
material should not be used beyond 6 months from the date of manufacture.

**MIL-L-23398 (ASG): Lubricant, Solid Film, Air Drying**

This specification establishes the requirements for an air-drying film lubricant intended to reduce wear and prevent seizing and galling. Condensed specification requirements, uses, and limitations are given below.

**Material.**—The mixture (in a spraying consistency) is to be finely powdered lubricating solids dispersed in a suitable binder. The applied film shall cure at room temperature in not more than 6 hr.

**Film condition.**—The bonded solid-film lubricant shall appear uniform in color; smooth; free from cracks, scratches, blisters, foreign matter, grit, rough particles, bubbles, pinholes, runs, sags, and other surface imperfections; and shall show no evidence of separation of ingredient.

**Film adhesion.**—The bonded solid-film lubricant shall not be lifted from a test panel by the pressure-sensitive masking tape method. A uniform deposit of powdery material may cling to the tape, but lifting of any flakes or particles which expose any bare metal shall indicate unsatisfactory adhesion. Test should be made in accordance with reference 137A.

**Thermal stability.**—The bonded solid film lubricant shall not flake, crack, or soften; and shall have satisfactory adhesion when tested for 3 hr at \(-65^\circ F\) and \(+500^\circ F\). Test should be made in accordance with reference 137B.

**Endurance life.**—The bonded solid-film lubricant, tested in the Falex Lubricant Tester, shall have an average life of not less than 60 min at 1000-lb gage load. The endurance life of any single run shall not be less than 50 min. Test should be made in accordance with reference 137C.

**Load capacity.**—The bonded solid-film lubricant, tested in the Falex Lubricant Tester, shall have a minimum load capacity of 2500-lb gage load (ref. 137D).

**Corrosion resistance.**—The bonded solid-film lubricant on anodized aluminum panels shall show or cause no discoloration, pitting, formation of white deposits, or other evidence of corrosion after 500 hr at 120°F and 95 percent humidity. Test should be made in accordance with reference 137E.

**Storage stability.**—This solid-film mixture shall remain in a homogeneous blend showing no evidence of gelation after storage in a closed container for 6 months at room temperature. After storage, the bonded solid-film lubricant must conform to the other requirements of this specification.
Uses.—This air-drying solid-film lubricant is intended for use on steel, titanium, aluminum, and aluminum alloys. It is useful where conventional fluid lubricants are difficult to apply or may be contaminated with dirt and dust. It is generally suitable for sliding motion surfaces, such as plain spherical bearings, tracks, hinges, and cams; and is recommended for use where solid film lubricants that require elevated-temperature cures cannot be applied because of material or other reasons.

Limitations.—This lubricant should not be used with oil and grease unless experience indicates otherwise. Application should be conducted in a well ventilated area where no flame or ignition sources are present. This material is not a substitute for MIL-L-8937 lubricants, as it has lower wear-life and load-carrying ability.

MIL-L-46010 (MR): Lubricant, Solid Film, Heat-Cured, Corrosion Inhibiting

This specification covers a resin-bonded, heat-cured, solid film lubricant intended to reduce wear, prevent galling and seizure, and provide corrosion protection to metals. This lubricant does not contain graphite or powdered metals. Condensed specification requirements, uses, and limitations are given below.

Material.—The lubricant shall consist of a dispersion of lubricative pigment or pigments in a thermosetting resin with or without additives.

Film thickness.—The lubricant shall be capable of being applied by brush, dip, or spray methods and cured to a film thickness between 0.0002 and 0.0005 in. All film measurements must be within these limits.

Wear-life.—The cured lubricant film shall provide an average minimum Falex wear-life of 450 min at 1000-lb gage load. No single test shall have less than a 390-min wear-life. A minimum of four tests is required in accordance with reference 137C.

Load-carrying capacity.—The film shall provide an average minimum Falex load-carrying capacity of 2000 lb gage. No single test shall have a load capacity of less than 1750 lb gage. A minimum of two tests is required in accordance with reference 137D.

Corrosion protection.—The film when applied to 3 × 6 in. steel-sheet (SAE 1009) test panels shall show a maximum of three rust dots per panel after a salt spray exposure of 100 hr. Test should be made in accordance with reference 137C.

Film adhesion.—The cured lubricant film shall not be lifted from the test panel by the pressure-sensitive masking tape method. A uniform deposit of powdery material may cling to the tape, but lifting of any flakes or particles which expose any bare metal shall
Indicate unsatisfactory adhesion. Test should be made in accordance with reference 137A.

**Fluid resistance.**—The film shall pass the film adhesion test after half immersion for 24 hr at 74° F in each of the following fluids: standard hydrocarbon test fluid, aviation gasoline, jet fuel, hydraulic fluids (petroleum and nonpetroleum base), aircraft lubricating oils (petroleum and synthetic base), silicone fluid, and trichloroethylene.

**High and low temperature stability.**—The film shall pass the film adhesion test after exposure to a temperature of 500° F for 4 hr, and a low-temperature cycle of 4 hr on a cake of dry ice (carbon dioxide) in accordance with reference 137B.

**Storage stability.**—The lubricant dispersion stored in a closed container for 6 months at room temperature shall meet the wear-life and corrosion-protection requirements of this specification.

**Uses.**—This resin-bonded solid film is intended for use on aluminum, copper, copper alloys, steel, stainless steel, and titanium. It is generally suitable for sliding motion operations involving such items as plain and spherical bearings, tracks, hinges, threads, and cam surfaces. The film is serviceable under the following conditions: where conventional lubricants are difficult to apply or retain, where other lubricants may be contaminated by dirt or dust, a temperature range between −65° F and +350° F; in mechanisms operated at infrequent intervals; and in mechanisms to be lubricated for life.

**Limitations.**—This film lubricant should not be used on materials adversely affected by the heat-cure cycles of 400° F for 1.0 hr or 300° F for 2 hr. It should be applied in well-ventilated areas where no flame or ignition source is present.

**MIL-L-81329 (WEP): Lubricant, Solid Film, Extreme Environments**

This specification establishes the requirements for a solid-film lubricant to be used in extreme environments (including temperatures from −300° to +1000° F, liquid oxygen, and vacuum), to reduce wear and prevent galling and seizing of metal surfaces. Condensed specification requirements, uses, and limitations are given below.

**Material.**—The lubricant (in a spraying consistency) shall consist of high-quality lubricating solids in a suitable binder. Organic materials are not suitable for this lubricant. The lubricant material shall be nonflammable when heated by a Bunsen burner flame. The applied lubricant film shall be capable of being cured ½ hr at 77° F, 2 hr at 180° F, and 2 hr at 300° F.

**Appearance and film thickness.**—The solid-film lubricant shall be free of surface imperfections and show no evidence of separation
of material ingredients; the finished film thickness shall be between 0.0002 and 0.0005 in.

Film adhesion.—The lubricant shall not be lifted from the test panel by the pressure-sensitive masking tape method. A uniform deposit of powdery material may cling to the tape, but lifting of any flakes or particles which expose any bare metal shall indicate unsatisfactory adhesion.

Thermal stability.—The bonded solid-film lubricant applied to 18–8 stainless steel panels and exposed to 1000°F for 3 hr followed by 1 hr at −300°F shall show no flaking, cracking, or softening.

Endurance life.—The lubricant shall have a minimum average life of 100 min on the Falex lubricant tester at 1000-lb gage load. A minimum of four tests is required, each test having a life of at least 80 min.

High-temperature performance.—The lubricant tested by the method and equipment described in Federal Standard No. 791, Method 333 (ref. 138), shall demonstrate a useful life of 500 hr at 750°F and 10 000 rpm continuous running on M–10 steel bearing, SAE 204 size with ABEC–3 tolerance.

Vacuum performance.—The solid-film lubricant shall be applied to an antifriction bearing and subjected to a vacuum of 1.0×10⁻⁸ torr at 1000°F and 1250 rpm. A 5.0-lb axial and a 3.0-lb radial load shall be applied to the bearing. The lubricant shall demonstrate a minimum life of 100 hr. Failure is indicated by a 20°F rise in temperature of the bearing case or a 50 percent increase in the power required.

Shock sensitivity with LOX.—The lubricant tested in accordance with U.S. Air Force Specification Bulletin 527 shall give no reaction in 25 test drops at an energy of 70 ft-lb. The lubricant shall be spray deposited and cured in test cups prior to testing.

Storage stability.—A closed quart container of the solid-film solution shall be stored at 77°F for 6 months. It shall then be mechanically agitated for 5 min, the container opened, and the lubricant examined for homogeneity. Cured solid-film specimens shall than pass the film adhesion, thermal stability, and endurance life tests.

Uses.—This solid-film lubricant is intended for use in oxygen systems; space vehicles; bearings; and other equipment where the environments of temperature, nuclear radiation, and vacuum will not permit the use of conventional lubricants or organic-bonded solid-film lubricants.

Limitations.—This solid-film lubricant should not be used on materials which may be adversely affected by the required cure temperature of 300°F. Also, it should not be used with oils or greases unless experience indicates otherwise.
APPENDIX A

MSFC–SPEC–253A: Lubricant, Dry Film, Ceramic, MLF–5 and MLF–9 (Preparation and Application)

This specification covers the requirements for the preparation of parts and application of two dry-film lubricating materials (designated MLF–5 and MLF–9) that have low friction and will support high loads. Surface finish and preparation for these solid film lubricants require a chemically and mechanically cleaned surface with a smooth dry-honed finish not exceeding 8 to 13 μ in. rms. Condensed specification requirements are given below.

MLF–5 solid film.—Preparation requires careful mixing of specified amounts and particle sizes of several solid-film powders, including: molybdenum disulfide, graphite, gold, and sodium silicate in specified proportions of distilled water. Powdered ingredients for this solid-film lube shall pass through a 325-mesh sieve (44 μ).

MLF–9 solid film.—Preparation requires careful mixing of specified amounts and particle sizes of several granular powders, including: molybdenum disulfide, graphite, bismuth, and aluminum phosphate, in specified proportions of distilled water. The powdered ingredients for this solid film shall pass through a 325-mesh sieve (44 μ).

Mixing, application, and cure.—Both MLF–5 and MLF–9 must be continuously stirred during mixing, and the mixed lubricant solution must also be stirred in the container during spray application. The solid film must be applied as a fine mist spray, using a dry-nitrogen pressure source. The rate of application should be such that the film appears to dry on contact and no wet spot should appear. The thickness of individual coats or layers of film lubricant should be between 0.0001 and 0.0004 in. Both MLF–5 and MLF–9 require sequential heat-cure cycles, the maximum for MLF–5 is 325° F; for MLF–9, the maximum is 460° F.

Workmanship.—When applied to parts and cured as specified, both MLF–5 and MLF–9 lubricants shall show no evidence of cracking, flaking, or other defects that adversely affect their intended use.

MSFC–SPEC–502: Lubricant, Dry Film, Ceramic, MLF–5 (Preparation and Application (Proposed))

This specification covers the requirements for the preparation of the parts and method of application of one type of LOX-compatible dry-film lubricating material (MLF–5) that has a low coefficient of friction and can support high loads without penetration.

Uses.—MLF–5 is intended for use in the space industry where low friction and LOX compatibility are prime requirements.

Limitation.—This material has a porous structure and does not protect the bearing surface from corrosion.
**MSFC–SPEC–50M60434: Lubricant Dry Film, MLR–2**  
*(Preparation and Application)*

This specification covers the requirements for the preparation of the parts and application of dry-film lubricant (MLR–2) that has low coefficients of friction and the capability of supporting high loads without penetration.

*Uses.*—MLR–2 is intended for use where long life of the bonded film is a requirement over wide temperature ranges and at high loads.

*Limitations.*—The material is not LOX-compatible and therefore should not be used in areas where LOX is present.

**MSFC–SPEC–81205: Bonded Solid Dry Film Lubricants, Material Specification for and Application of Bonded Solid Dry-Film Lubricants**

This specification covers the quality of the solid-lubricant materials as well as the application procedures.

There are several types of dry lubricants included in this specification: type I for general purpose use from −100°F to +475°F; type II cryogenic, wide temperature range for operation at −150°F to +1100°F; and type III cryogenic, wide temperature range and LOX-compatible for operation from −320°F to +650°F.

**MIL–G–21164: Grease, Molybdenum Disulfide, for Low and High Temperatures**

This specification covers one grade of grease intended for use as a lubricant on heavily loaded sliding steel surfaces. It is composed of an hydroxystearate soap and 5.5 percent by weight of molybdenum disulfide as an extreme pressure agent.

*Uses.*—This grease is intended for use as a lubricant for accessory splines, heavily loaded sliding steel surfaces, or for antifriction bearings carrying high unit loads and operating through wide temperature ranges where molybdenum disulfide will prevent or delay seizure in the event of inadequate lubrication.

*Limitations.*—This grease shall not be used for other than steel surfaces without prior performance evaluation.

**MIL–L–25681 C: Lubricating Oil, Molybdenum Disulfide, Silicone Base, for High Temperatures**

This specification covers one grade of lubricant for use on sliding surfaces subject to high temperatures in aircraft gas-turbine engines. The material is a composition of 50 percent by weight of molybdenum disulfide (MIL–M–7866), and 50 percent by weight of methyl phenyl polysiloxane, silicone fluid.
Uses.—This material is intended for use on slow-speed sliding surfaces operating at temperatures up to $750^\circ$ F and for use as an antiseize compound on threaded parts which operate at temperatures to $1400^\circ$ F.

Limitations.—This material should not be used on antifriction bearings under any circumstances.

MIL-B-8942: Bearings, Plain, TFE Lined, Self-Aligning, Self-Lubricating

Uses.—This specification covers plain spherical bearings which are self-aligning and self-lubricating by incorporating TFE in a liner between the inner ring (ball) and outer ring (race) for use in the temperature range $-65^\circ$ to $+250^\circ$ F.
Contact Stresses in Wear-Life Testers—Dual Rub-Shoe, Falex, and Pin-on-Disk Machines

In the development and evaluation of solid-film lubricants, it is often desirable to know the stresses to which films are subjected during friction and wear tests. A knowledge of the stress level in the various contacts found in different test devices provides a better basis for comparing test results than the applied load. The purpose of this appendix then, is to determine the contact stresses that are encountered in the testing of various materials and lubricants on three types of friction and wear-life test devices. The different materials used in the calculation of the stresses were chosen because of their widely varying moduli of elasticity.

Contact stresses result from the pressure of one elastic solid on another at limited areas of contact. Most load-resisting members are designed on the basis of stress in the main body of the member, that is, in the portions of the body not affected by the stresses at and near the surface of contact where the loads are being applied. In some members, however, the stresses resulting from the surfaces of two bodies being forced together by external loads turn out to be the significant stresses. These stresses occur on or slightly beneath the surface of contact and often are the cause of failure in one or both of the bodies. Examples where the contact stresses may well be the significant stresses are (1) at the area of contact between a roller or a ball and its race in a rolling element bearing and (2) at the area of contact between the teeth of a pair of gears in mesh.

The theory used in the determination of contact stresses was first set forth by H. Hertz in 1881; consequently, these stresses are commonly called Hertzian contact stresses. The theory and equations used in this work are an adaptation of Hertz’s (ref. 139).

The following notation and interpretation of terms are common to the calculations for all testing devices:

\[ P = \text{total force exerted by body 1 on body 2, and vice versa} \]
\[ E_1, E_2 = \text{moduli of elasticity for bodies 1 and 2} \]
\[ \mu_1, \mu_2 = \text{Poisson’s ratio for bodies 1 and 2} \]
$R_1, R_1' = \text{minimum and maximum numerical values of radii, respectively, of the surface of body 1 at the point of contact}.$ (The signs of $R_1$ and $R_1'$ are: if the center of curvature is inside the body, the radius is positive; if outside the body, it is negative.)

$R_2, R_2' = \text{same as } R_1, R_1', \text{ for body 2.}$

**TEST DEVICES AND CONFIGURATIONS**

The testing devices for which stresses were calculated are (1) the dual rub-shoe machine, (2) the Falex tester, and (3) the pin-on-disk apparatus. The dual rub-shoe machine and Falex tester involve line contact, that is, the contact of a circular cylinder resting on a plane. The pin-on-disk apparatus involves point contact which results from a sphere at rest on a plane. The maximum compressive stress is calculated for each of these three devices. The compressive stress was chosen because it is the maximum contact stress. The effect of any lubricant film has been neglected in the calculations.

The rub-shoe machine uses a specimen configuration consisting of two opposed $\frac{1}{4}$ in. wide rub shoes loaded against a $1\frac{3}{8}$ in. diam by $\frac{3}{8}$ in. thick rotating lubricated disk as shown in figure 65. The rub-shoe machine is used primarily for determining wear-life and friction of solid-lubricant films.

The Falex tester uses a specimen configuration consisting of a $\frac{1}{4}$-in. diam rotating pin clamped between two $\frac{1}{2}$-in. diam and $\frac{3}{8}$ in. long V-shaped blocks in a nutcracker type arrangement so that load is applied to the blocks and consequently to the rotating pin. This configuration is shown in figure 66. The Falex tester is used mainly for evaluating the load-carrying capacity of a lubricant.

The pin-on-disk configuration consists of a $\frac{1}{4}$-in. diam dry ball sliding on the lubricated flat surface of a rotating disk shown in figure 67. This apparatus is used to determine the wear-life characteristics of solid-film lubricants.

In calculating the contact stresses for the three devices, these material combinations were assumed: (1) steel against steel, (2) bronze against bronze, and (3) molybdenum against molybdenum.
Friction values ranging from 0.00 to 0.20 were used in the calculations for rub-shoe and Falex devices. Friction was not considered in the calculations for the pin-on-disk apparatus because its effect on point contact loadings has yet to be determined. Moduli of elasticity equal to 15, 29, and 50 million psi were used, respectively, for bronze, steel, and molybdenum. Values of Poisson's ratio of 0.35 for bronze and 0.25 for both steel and molybdenum were obtained from Mark's Mechanical Engineers' Handbook (ref. 140) and were used in the calculations.

Sample stress calculations and derived equations for the maximum compressive stress $S_c$ are presented for each of the three testing devices. Computer programs for each of the three devices were written for the purpose of calculating $S_c$ for different values of load and friction. Computer plots were then made of stress vs load with different values of friction for the three material combinations of the rub-shoe and Falex devices. Plots were also made of stress vs load for each of the three devices to show the effect of the modulus of elasticity on stress (figs. 68 through 76).
HERTZIAN CONTACT STRESS
RUB-SHOE TEST SPECIMEN, STEEL ON STEEL
F IS FRICTION COEFFICIENT
F=0.75
F=0.15
F=0.10
F=0.05
F=0.0

FIGURE 68.—Effect of load and friction on contact stress —rub-shoe configuration, steel on steel.

HERTZIAN CONTACT STRESS
RUB-SHOE TEST SPECIMEN, BRONZE ON BRONZE
F IS FRICTION COEFFICIENT
F=0.20
F=0.15
F=0.10
F=0.05
F=0.0

FIGURE 69.—Effect of load and friction on contact stress —rub-shoe configuration, bronze on bronze.

STRESSES AND THEIR SIGNIFICANCE

In the examples used, the stress calculated was the principal or maximum compressive stress. For two normally loaded bodies in point or line contact, the compressive stress is the maximum stress and is always located at the surface of contact. Shear stresses are also present and are located at a point beneath the surface of contact. The presence of a frictional force causes the maximum values
of the compressive and shear stresses in the two elastic bodies to become substantially larger than those produced by the normal force alone. Furthermore, the addition of a frictional force to a normal force causes a change in the shear stresses in the region of the contact surface. One important change is that the location of the point at which the maximum shear stresses occur moves from beneath the surface of contact towards the surface of contact. In fact, when the
coefficient of friction is 0.1 or greater, this point is located at the contact surface. Shear stresses are significant because engineers usually attribute failure resulting from cracking, pitting, etc., in ductile materials to shear stresses. The theory and means for calculating shear stresses can be found in reference 140.

The determination of contact stresses provides a basis for comparing the relative severity of various test devices and their partic-
ular test conditions on different materials and lubricants. Plots of stress vs load in this report assume bare metal contact and represent maximum values. From these plots one can see that the maximum contact stress is not only affected by load, but is also affected by friction and modulus of elasticity. The introduction of a lubricant film between contact surfaces also affects the contact stresses. In this report, however, it was neglected because the theory required for including the film effect on contact stresses has not yet been
developed. Nevertheless, its effect would probably be twofold: (1) the contact stresses would be decreased because of the load distributing effect of a film and (2) the contact stresses would be decreased because of lower friction between the two bodies.

**SAMPLE STRESS CALCULATIONS AND STRESS VERSUS LOAD PLOTS**

**Rub-Shoe Machine Calculations**

Calculation of stresses for the rub-shoe machine with a material configuration of steel on steel and various values of friction was made as follows.

The following terms and values were used:

\[ P = \text{rub-shoe load, lb} \]

\[ \mu = \mu_1 = \mu_2 = \frac{1}{4} \]

\[ E = E_1 = E_2 = 29 \, 000 \, 000 \, \text{psi} \]

\[ R = 1\frac{1}{10} \, \text{in.} \]

\[ f = \text{coefficient of friction, values ranging from 0.00 to 0.20} \]

The equation of maximum compressive stress is

\[ S_c = \left[ 1.0 + 1.11(f) \right] b/\Delta \]

where \( b \) and \( \Delta \) are variables dependent on the particular physical configuration.

The equation for \( \Delta \) is
\[ \Delta = 4R \left( \frac{1-\mu^2}{E} \right). \]

In evaluating \( \Delta \),
\[ \Delta = 2.75 \left( \frac{1^{1/6}}{29 \ 000 \ 000} \right) = 8.8 \times 10^{-3} \text{ in}^2/\text{lb}. \]

The equation for \( b \) is
\[ b = \sqrt{2q \Delta / \pi} \]
where \( q \) is the load per unit length of the contact area.

In evaluating \( b \),
\[ b = \sqrt{2q \Delta / \pi} = 4.74 \times 10^{-3} \sqrt{P} \text{ in.} \]

The value of the ratio \( b/\Delta \) is
\[ b/\Delta = 4.74 \times 10^{-3} \sqrt{P} / 8.8 \times 10^{-3} = 0.538 \times 10^4 \sqrt{P} \text{ psi.} \]

With the above values, an equation for \( S_c \) can be written in terms of \( P \) and \( f \) as
\[ S_c = \left[ 1.0 + 1.11(f) \right] 0.538 \times 10^4 \sqrt{P} \]
\[ = 5380 \times \sqrt{P} + 5970 \times f \times \sqrt{P} \text{ psi.} \]

As a comparison, the equation for the maximum shear stress is
\[ S_s = 0.43 \left( \frac{b}{\Delta} \right) \]
\[ = 0.43 \left( 5380 \times \sqrt{P} \right) \]
\[ = 2320 \sqrt{P} \text{ psi.} \]

**Falex Tester Calculations**

Stress calculations for the Falex tester are very similar to those for the rub-shoe machine; the only differences are that a material combination of bronze on bronze is used and the following terms are changed to:
- \( P \) = jaw load, lb
- \( \mu = \mu_1 = \mu_2 = 0.35 \)
- \( E = E_1 = E_2 = 15 \ 000 \ 000 \text{ psi} \)
- \( R = \frac{1}{8} \text{ in.} \)

Thus the equation of \( S_c \) is
\[ S_c = 5580 \sqrt{P} + 6220(f) \sqrt{P} \text{ psi.} \]

**Pin-on-Disk Calculations**

Stress calculations for the pin-on-disk apparatus with a material combination of molybdenum on molybdenum proceeded as follows
Using the following terms and values of

\[ P = \text{contact load, lb} \]
\[ \mu = \mu_1 = \mu_2 = \frac{1}{4} \]
\[ E = E_1 = E_2 = 50\,000\,000 \text{ psi} \]
\[ R = \frac{1}{8} \text{ in.} \]

The equation of maximum compressive stress is

\[ S_c = 0.64 \frac{b}{\Delta}. \]

The equation for \( \Delta \) is

\[ \Delta = \frac{2}{A+B} \left( \frac{1-\mu^2}{E} \right) \]

where \( A \) and \( B \) are constants depending on the radii of the two bodies in contact; and in this case are both equal to four.

In evaluating \( \Delta \),

\[ \Delta = \frac{2}{8} \left( \frac{11/4}{50 \times 10^6} \right) \]
\[ = 0.0047 \times 10^{-6} \text{ in.}^2/\text{lb}. \]

The equation for \( b \) is

\[ b = 0.90 \sqrt{P \Delta} \text{ in.} \]

In evaluating \( b \),

\[ b = 0.90 \sqrt{P(0.0047 \times 10^{-6})} \]
\[ = 0.150 \times 10^{-2} \times \sqrt{P} \text{ in.} \]

Using these values for \( b \) and \( \Delta \), \( S_c \) becomes

\[ S_c = 0.64 \frac{b}{\Delta} \]
\[ = 0.64 \left( 0.150 \times 10^{-2} \sqrt{P} / 0.0047 \times 10^{-6} \right) \]
\[ = 204 \, 260 \times \sqrt{P} \text{ psi.} \]

**Stress Versus Load Plots**

The plots of stress vs load for all three testing devices and all three material combinations are as shown in figures 68 through 76.
Glossary

Antifriction bearing—usually denotes a ball or roller bearing.

Binder—the material used to hold the pigment (lubricant) of a solid lubricant to the substrate.

Cold flow—a characteristic of plastic materials whereby they flow out of a high-load area at room temperature.

EP additives (extreme pressure additives)—substances added to oils and greases to prevent metal-to-metal contact in highly loaded areas. In some cases this is accomplished by using additives which react with the metal to form a protective coating.

Free acid points—titration of a phosphatizing bath sample to a methyl orange end point. The point range is from zero (weakest acid) to 14 (strongest acid).

Hard vacuum—a term used to denote a very low pressure.

Impact sensitivity—the tendency of some materials to react with liquid oxygen when subjected to mechanical impact or vibration. This reaction is often explosive in nature.

LOX—liquid oxygen.

Lubricity—the property of forming a lubricating film between moving surfaces, particularly when such surfaces are subject to heavy loads and rapid movements. Lubricity depends partly upon the wetting ability of the film-forming material. Oiliness is sometimes used as approximately equivalent to lubricity.

Pigment—the lubricating material (such as MoS₂ and graphite) used in a solid lubricant.

Pretreatment—usually refers to the treatment of a substrate prior to the application of a solid-film lubricant.

PV—the product of the unit load (psi) based upon a projected bearing area (in.²) and the linear shaft velocity V (ft/min).

Salt base—the hydrolysis product of an alkaline material and a silicon halide.

Solid lubricant—a solid material that provides lubrication between two relatively moving surfaces.

Stick-slip—a phenomenon found in friction and wear investigations. In this phenomenon, the relative motion between two surfaces need not be a continuous process but may proceed in a series of intermittent jerks, friction building up during the stick process and falling off during slip.

Synthetic lubricant—generally a nonpetroleum base lubricant; a lubricating material made synthetically, usually with a very narrow boiling range.

Polytetrafluoroethylene (PTFE)—a plastic material used as a solid lubricant.

Timken ring—a standard ring used on several bench-type wear and friction testers. It is the registered trademark of the Timken Bearing Co., Canton, O.

Torr—the unit of pressure adopted by the American Vacuum Society. It is defined in terms of a standard atmosphere which is now taken as 1 013 250 dynes/cm². The torr is 1/760 atm or 1340 dynes/cm². One torr is approximately equivalent to 1 mm Hg.

Wear-factor (K)—a proportionality constant relating radial wear R to load P, velocity V, and time T in the equation R = KPTV.
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B. Method 3805—Thermal Stability of Dry Solid-Film Lubricants.
C. Method 3807—Endurance (Wear) Life of Dry Solid-Film Lubricants.
D. Method 3812—Load-Carrying Capacity, Dry Solid-Film Lubricants.
E. Method 4001—Protection, Salt Spray (Fog).

