

SURVEY OF SOLID FILM LUBRICANTS

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ABSTRACT

Discussion of the capabilities and availability of solid film lubricants is presented. Solid film lubricants available from commercial vendors are of the organic resin-bonded type and are temperature-limited to about 450°F because of the organic resins. Most films contain molybdenum disulphide or graphite pigment. Results indicate these films are capable of 70 hours continuous operation at ambient temperatures, 80,000 psi load and speed to 200 ft/min.

Various test devices are discussed as to capabilities, limitation, etc. Results are presented for various operating conditions.

Research in high-temperature candidate films is discussed in detail. Problem areas such as binders, temperature dependency of some of the pigments, etc., are outlined. Methods and criteria for selecting possible materials as solid film lubricants are stated. Design and development of wear life testing machines for high-temperature and vacuum conditions is presented along with results of many tests conducted on these films under various conditions.

Results to date indicate lead sulphide (PbS) bonded with boric oxide (B_2O_3) is the best solid film lubricant for use at 1000°F. Comparative tests conducted on the Hohman A-6 Tester indicated a wear life of approximately 14,000 feet, at 1000°F, 100-pound load and 126 ft/min sliding speed. A conventional film on this tester gives an average wear life of approximately 40,000 feet, at 250°F, 200-pound load and 126 ft/min sliding speed. This film suffers very low wear life at temperatures below 800°F. This behavior is receiving extensive investigation at the present time. Initial vacuum studies have been conducted and preliminary results are discussed.

The need for a more fundamental knowledge of the mechanisms involved with these materials is pointed out. Present programs in this area are discussed briefly along with future thinking to bring the fundamental research up to that of the present developments.

INTRODUCTION

This presentation will survey the general area of solid film lubrication and attempt to depict the current developments, especially in the high-temperature regions. Changing Air Force lubrication requirements have generated the need for lubricating media beyond the capabilities of well known grease and oil systems. The requirements for advanced weapon systems include reliable operation for long periods of time over a wide range of environmental conditions. These conditions include wide temperature ranges with operation up to and including 2000°F as well as environments of nuclear radiation and vacuum.

As will be seen later in the presentation, solid film lubricants are currently in wide use in various military vehicles with applications being found in slow sliding bearings operating under loads of 2000 to 80,000 psi in the temperature range of ambient to 450-500°F. Of course, one may say that greases and oils will operate satisfactorily under these conditions. This is true; however, one of the ultimate goals in using solid films is lubrication for the life of the part. This will eliminate the need for costly periodic maintenance in which it is necessary to replenish the oil or grease at regular intervals to insure proper operation. Specific applications in use now include lubrication of outer control surfaces on aircraft which are practically impossible to lubricate with conventional lubricants. It is anticipated that as advanced flight vehicles become more and more complex in design and operate in higher temperature ranges, these solid film lubricants will demonstrate their capabilities for providing satisfactory lubrication.

Since there currently exist different concepts as to how to overcome the high-temperature lubrication problems, it would be worthwhile to define the term "solid film lubricant" and to differentiate this term from other friction and wear reduction techniques. The term, as used throughout this presentation, will be to designate a series of lubricating materials consisting of a friction and wear reducing pigment, such as graphite, molybdenum disulphide, or other metal salts, bonded to the bearing surface with some type of adhesive. Most current films use organic resins as adhesives while development work in the 1000 to 1500°F range is considering ceramic type materials. Another term often used synonymously with solid films is that of "dry film lubricants." This term, for the purposes of this presentation, will be used to refer to materials which chemically react with the metal surface or are plated on to provide wear resistance. These materials are usually very hard (such as titanium carbide) and offer very high thermal stability. Hence, they are being considered for high-temperature bearing operation, but cover an area of wide scope of their own and will not be reviewed in this presentation.

The discussion which follows will be essentially divided into three areas. The first will cover presently available films of the organic resin type. Present uses and test results will be presented. The second area will describe the development effort currently being conducted for operational films in the 1000 to 1500°F range. Problem areas and related research programs will be discussed. The third area will be a discussion of anticipated future requirements and approaches to be considered.

PRESENTLY AVAILABLE SOLID FILMS

As was stated earlier, there are several commercially available solid film lubricants which are seeing extensive use in the aircraft industry as well as non-military application. These films are composed of a lubricating or friction reducing pigment, a bonding agent is, in most cases, an organic adhesive, and a thinning solvent to give the proper consistency for application. This mixture is applied by brushing, dipping or spraying to a metallic bearing surface which has been pretreated in some fashion to increase the adhesive bond between the metal and film. Of the above three methods of application, spraying is used almost entirely because of the control of uniformity in film thickness. At the present time there are some ten to twelve major commercial vendors for these films. The relative merit of one film over that of another depends not only on the vendor to whom you are talking, but also in the pigments selected, the binders used, the ratio of the two to each other, etc., in a cured composition. Many of the films are very similar in composition.

The lubricating pigments used in these films are almost entirely graphite and molybdenum disulfide. Some of the films are composed of one or the other of these pigments, while others contain a mixture of the two. In the case of the mixture, the practice has been to use approximately eighty to ninety percent molybdenum disulfide and the remainder graphite. The contributing factor of this small percentage of graphite has not been fully explained, but its effectiveness has been demonstrated in bench evaluation and service application.

Bonding agents consist primarily of organic resins and inorganic adhesives. The organic resins are of the phenolic and epoxy resin type and are used to a greater extent in these particular films than are the inorganic binders. The epoxy compositions are preferred over the phenolic; however, in a few cases, mixtures of the two have been utilized. These organic resins are the

limiting factors for operation of these films above 450°F. At this temperature thermal degradation begins and wear life falls off rapidly with increasing temperature. The operating temperature of these films is extended with the use of an inorganic adhesive or binder such as a metal matrix composition or ceramic. Using these binders, operation at higher temperatures is primarily limited by oxidation of the lubrication pigment; molybdenum disulfide suffers severe oxidation at temperatures above 750°F while graphite is stable to approximately 1000°F, but fails due to a loss of absorbed water.

Thus far only the lubricating pigments and bonding agents have been discussed. What then is to be said about the bearing material or metal surface to which these lubricant-binder combinations will be applied? It has been shown that the choice and controlled application of the proper pretreatment on the substrate metal will increase wear life performance many fold. (1, 2) Since surface pretreatment is almost as important as the film composition, it is wise to consider the various possibilities. Surface treatments commonly used for ferrous metals include vapor and grit blasting, iron and manganese phosphating. The phosphate treatment has shown the greatest influence in increasing wear life, but unfortunately is limited to metallic substrates of iron and steel. The phosphate pretreatment creates a non-metallic coating consisting of iron and manganese phosphates deposited in a crystalline form on the substrate metal. This surface serves to provide a stronger bond between the lubricant-binder composition and the metal surface. The grit and vapor blast are purely mechanical workings of the metal surface to create a roughened area allowing the binder material more surface area contact which increases adhesion. The phosphating procedure requires another serious consideration of its method of application. Bath and solution control probably have the greatest influence on phosphating iron and steel surfaces. (1) Temperature must be controlled within 5 to 6°F of the optimum temperature in order to assure proper crystal deposition. The mechanism of the kinetics of this pretreatment are not completely understood nor defined chemically. Therefore, it is impossible to determine the proper bath conditions except through a trial and error procedure. The rigid control of the bath conditions is believed to be one of the major factors in the wide variance of wear life from batch to batch or sometimes even within a batch. It is generally agreed that the closer the bath conditions are controlled, the better the films will be with more consistent results.

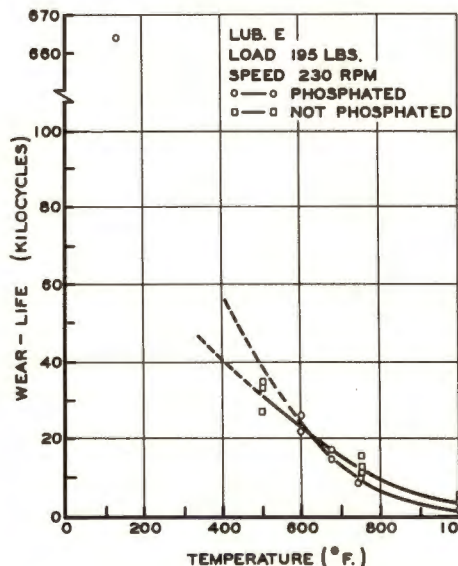


FIGURE 1. WEAR LIFE VS. TEMPERATURE

One other deficiency in the phosphate pretreatment is its temperature limitation. The films that are stable above 600°F have their optimum performance limited to this temperature if they have a phosphate pretreatment. Figure 1 shows data typical of this reduction in endurance life. (3) As shown in this figure, the cross-over point for phosphated and non-phosphated surface pretreatments is about 600°F. The film used in these tests is a commercially available metal-matrix bonded molybdenum disulfide lubricant. Low-temperature tests are not shown on this chart but have been run. The dotted portion of the curve indicates the trend to be expected with decreasing

temperature. At room temperature, results on the film with a phosphate pretreatment have given satisfactory results above 100,000 cycles under the same test conditions. It appears, therefore, that since phosphating is unsatisfactory above 600°F, mechanical working of the surface as with grit or vapor blast will have to be utilized or a better high-temperature pretreatment developed.

Now that the basic thinking that goes into the preparation of these films has been discussed, the next step would be to discuss the effects of various environments on the solid film lubricant itself. A great deal of work has been conducted on these commercial films as well as research compositions. Before discussing the results, it is perhaps wise to describe briefly the testing equipment in order to have a common understanding of some of the evaluation tests. The best test of any lubricant would be one conducted on the component or bearing in question, but this often proves too expensive and time consuming. Most of the evaluations are performed with devices which attempt to simulate the various environmental conditions to be encountered. Such techniques allow simple test specimens and a greater number of tests to be conducted. These machines evaluate the relative merits of one film to another under the desired test conditions and report some parameter such as endurance time to failure. Wear life of a certain number of hours for a particular test on a film does not mean that the film will perform in the same manner in an actual application. One may assume, however, that if it is better than another film under the same conditions, this relative relationship will probably hold in the actual usage.

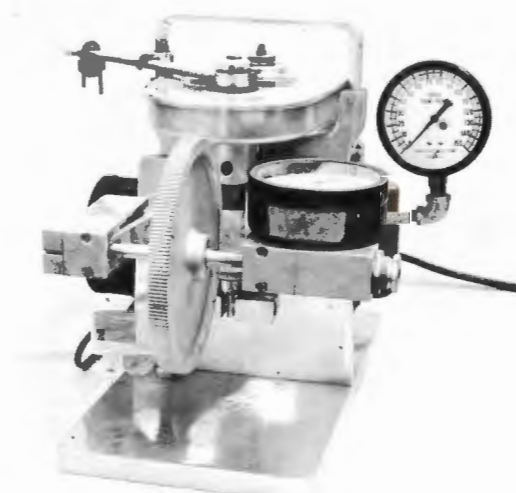


FIGURE 2. FALEX LUBRICANT TESTER

Probably one of the most familiar and most widely used pieces of apparatus of this type is the Falex lubricant tester. The Falex uses as test specimens coated pins 1/4" in diameter rotating between two coated V-blocks loaded to the desired test conditions through a jaw lever arm system. The standard Falex configuration is shown in Figure 2. A schematic of the test area is shown in Figure 3. Tests on this machine may vary from several minutes to two or three hours depending upon the film and test conditions studied.

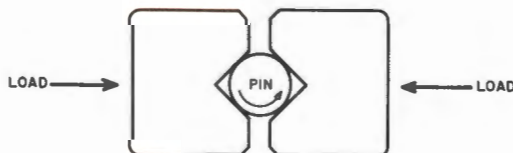


FIGURE 3. FALEX LUBRICANT TESTER, PRINCIPLE OF LOADING AND OPERATION

Another test apparatus commonly used employs a block sliding on a rotating cup. Many variations of this tester exist, but all use the same principle. The basic tester uses the outer race of a Timken bearing as a wear surface to which the solid film lubricant has been applied.

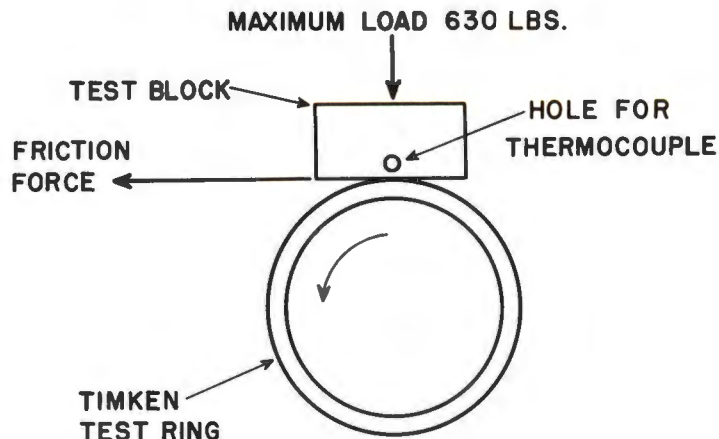


FIGURE 4. TIMKEN LUBRICANT TESTER

The test cup is rotated unidirectionally at a constant speed in contact with a rub shoe affixed to a lever loading apparatus and loaded to the desired level. As with the Falex tester, modifications have been made in the loading system and torque instrumentation to provide greater reproducibility in testing conditions, but the basic movement remains essentially the same. Figure 4 shows a schematic of the test area with the rub shoe and rotating cup. Figure 5 is the Alpha lubricant tester, one modification of the basic test. Neither the standard Falex nor the Alpha tester, as shown are capable of operation at extreme temperatures nor under unusual environments. Modifications have been made by research groups interested in environmental capabilities of dry films. The cup and shoe form of movement has been utilized on a number of new testers, some of which will be described later.

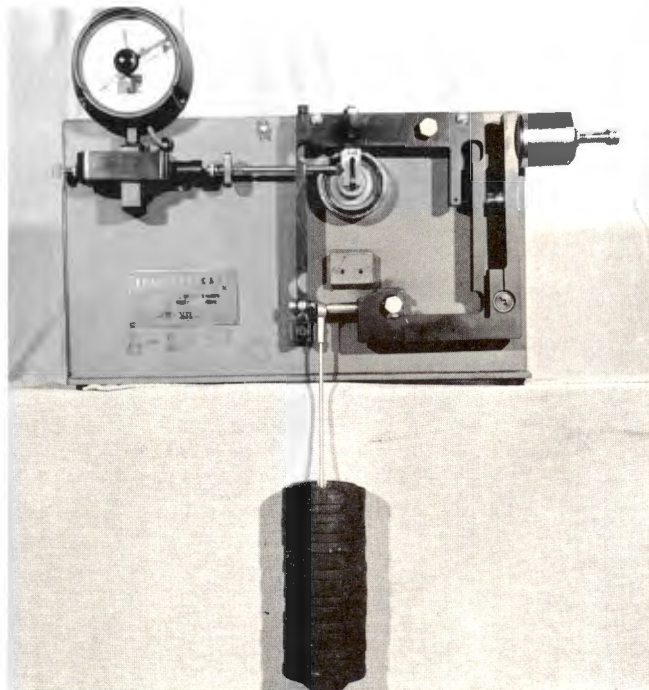


FIGURE 5. ALPHA LUBRICANT TESTER

One of the programs conducted at WADD on a modified Falex tester, was the evaluation of solid film lubricants at cryogenic conditions. For this study a standard Falex tester was modified to allow submersion of the test specimens in liquid nitrogen (b.p. -196°C). Figure 6 shows the modifications of this tester. Instrumentation included a recording wattmeter to monitor torque and a temperature recorder to indicate specimen temperature. Evaluation of films from seven commercial vendors plus two experimental V-blocks fabricated at WADD was conducted on this

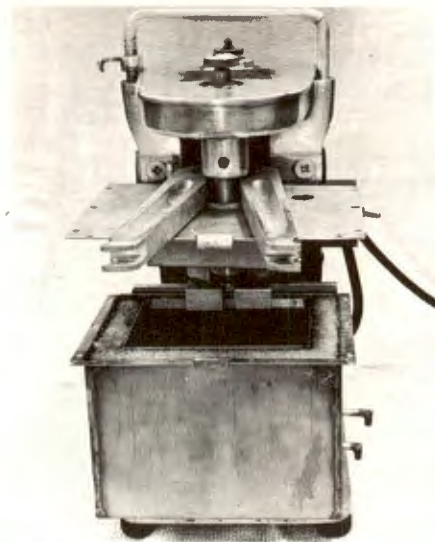


FIGURE 6. FALEX TESTER MODIFIED FOR CRYOGENIC ENVIRONMENT

test apparatus. This work has just been completed and will be published as a WADD Technical Report very shortly. Therefore, only a small portion of the data will be shown here to indicate the behavior of these films under the above environmental conditions. Table 1 shows the typical results obtained for some of these films. Basically, the results indicated that an 85 to 95 percent decrease in wear life could be expected in changing the environment from room temperature to that of liquid nitrogen. The films selected for this study represented a good cross section of commercial films including metal matrix, organic and inorganic films with pigments of molybdenum disulphide, graphite, combinations of these two, and films of polytetrafluoroethylene (Teflon). No particular lubricant shows favorable wear life in these conditions over that of another. The proposed mechanism of failure was mechanical rupture of the bond between metal and film due to the initial thermal shock. NASA has shown in their work on dry lubricants in liquid nitrogen that polytetrafluoroethylene films provide exceptionally good lubrication at these conditions under light loads and relatively high sliding velocities.⁽⁴⁾ Apparently the properties of Teflon which cause it to fail at higher loads do not affect its lubrication at the cryogenic temperatures and light loads.

TABLE 1. PERFORMANCE OF SOLID FILMS IN LIQUID NITROGEN

Wear Life in Minutes in Falex Tester at 1000 Lb Load

	<u>Avg. Four Tests</u>	<u>Avg. Loss in Life, %</u>
Film A		
Room Temperature	206	88
Liquid N ₂	24	
Film B		
Room Temperature	421	96
Liquid N ₂	17	
Film C		
Room Temperature	296	97
Liquid N ₂	10	
Film D		
Room Temperature	437	96
Liquid N ₂	20	

Another program recently completed at WADD was the determination of the effects of speed and load on wear life of these commercially available films. In these tests a tester utilizing the rub shoe and rotating test cup was employed. This tester is one of the new wear testers available and known as the Hohman A-6. This design uses two rub shoes diametrically opposed which eliminates the problem of shaft deflection under high loads. Figure 7 shows the Hohman A-6 tester. Its capabilities are 1500°F temperature, light and heavy loads, and speeds from 50 to 800 rpm. In addition, the test area is housed in an environmental chamber capable of handling unusual corrosive contaminants, as well as pressures above and below ambient. The speed and load tests were con-

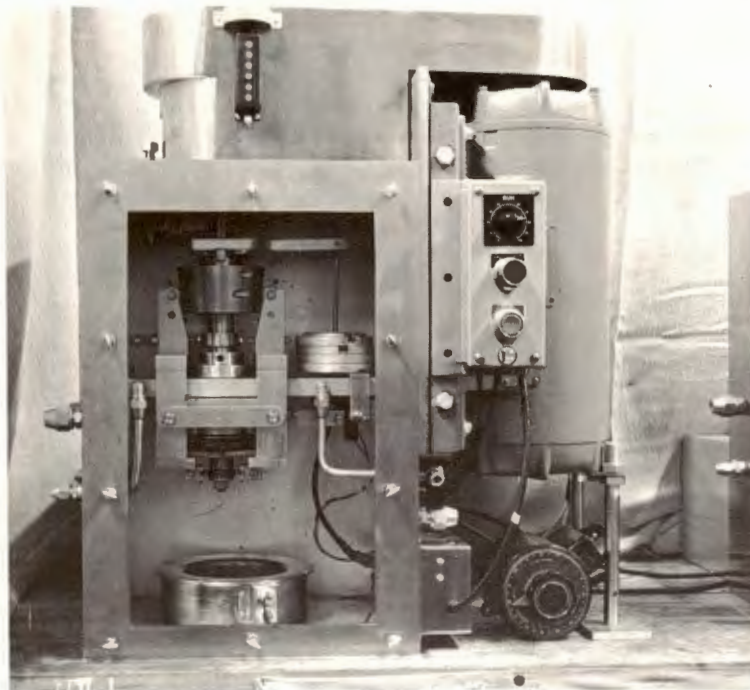


FIGURE 7. HOHMAN A-6 LUBRICANT TESTER

ducted on this machine because of its versatility and ease of changing test conditions. The tests were conducted at a constant temperature. A temperature of 250°F was selected because it falls well within the capabilities of the films and represented a mean temperature between ambient and the upper limit for these films. Tests were conducted under high and low speeds and loads, with intermediate speeds and loads as check points. Five commercial vendors participated in this program by submitting prepared test cups of two different formulations. Figure 8 indicates the

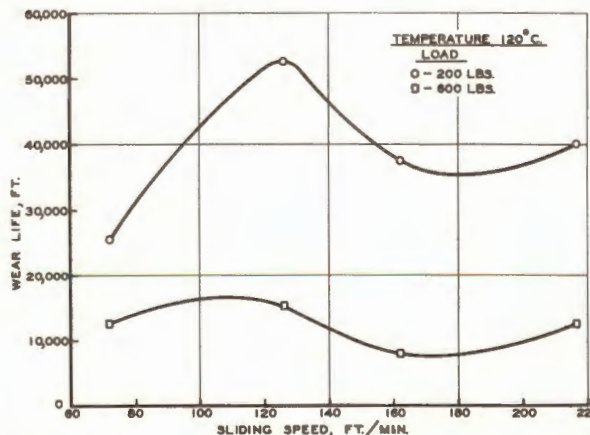


FIGURE 8. WEAR LIFE OF RESIN-BONDED FILMS VS. SPEED AND LOAD

typical data observed for these films under the above conditions. These data are being analyzed statistically to determine which of the above conditions has the greatest influence on wear life. The report describing the above program in detail will be published shortly.

Other programs conducted at WADD include effects of nuclear radiation on the performance of these films.^(2, 5) This work was reported at the last Lubricants Conference and will not be covered here. However, it was observed that radiation in general had very little effect either on the wear life of these films, or on the corrosion resistance, fluid resistance and thermal stability.

Qualification testing of these films under MIL-L-25504 has been conducted for over three years at WADD. Wear life is determined on the Alpha tester described earlier. Results obtained over this period of time have shown that present solid film lubricants should be capable of an average of 70 hours continuous operation. Test conditions have been 630 pounds normal load, unidirectional motion of 72 rpm and ambient room environments.

The commercial vendors are of course constantly striving to produce better films through their research and evaluation programs. Results of these tests are made public from time to time and present a fairly accurate picture of the capabilities of commercial solid film lubricants.

PRESENT HIGH-TEMPERATURE RESEARCH EFFORTS

Since standard lubricating techniques and present solid film lubricants will not meet the current projected high-temperature applications, research is necessary to provide new materials. Environmental conditions are so severe and varied that almost all organic materials will be destroyed through various mechanisms, thus the demand for lubricating systems employing materials stable under these environments is essential.

The types of motion and application for which these films are intended will include sliding bearings such as plain spherical, rod-end, ball sockets, ball and roller bearings both large and small, ball and screw actuators and many more. Almost all the forms of motion encountered by lubricants are included in the applications above. The need for solid films in each of these applications has been observed and undoubtedly will increase.

The environmental conditions which any one or all of these bearing systems will encounter include extremes in temperature from cryogenic applications to those in excess of 1500°F, hard vacuums as may be encountered in space, and nuclear irradiation at both high and low flux levels for varying periods of time. Atmospheres surrounding the bearings may consist of inert gases, propellant exhaust gases, and oxidizing conditions. It is unlikely that a given solid film lubricant will be applicable to all of these conditions and therefore solid film lubricants quite likely will be tailored to each specific application.

In the area of sliding applications at higher temperatures (700-1000°F), effort has been centered on inorganic pigments and ceramic or glass type binders. NASA, NAMC, MRI, WADD, the aircraft companies and solid film industry have all conducted work in the above areas.

NASA⁽⁶⁾ was among the first to conduct work on materials usable to 1000°F. Their most promising film consisted of lead oxide (PbO) bonded to the surface with sodium silicate (NaSiO₂). This film has shown low friction values at 1000°F but increases in friction at low temperatures. Equipment used by NASA in testing this film was a simple friction device consisting of a spherical rider rubbing on a flat surface. Evaluation of this film with a rub shoe and rotating cup tester indicates relatively higher wear compared to other high-temperature films.

Much of the initial high-temperature work at WADD was conducted by Midwest Research Institute under contract.⁽³⁾ A survey of over 22,000 compounds has been conducted by MRI⁽⁷⁾ which found approximately 1500 materials with melting points over 1000°F. From these materials approximately sixty solids were selected as possible lubricating pigments.⁽³⁾ Table 2 shows the criteria used to further screen these candidate materials.

Earlier work conducted by NACA indicates that the chlorides, oxides, and sulfides of metals in periods 3, 4, and 6 of the periodic table exhibit good lubricating characteristics.⁽⁸⁾ The selection of these additional criteria reduced the number of possible pigments to approximately 16 compounds. Since these lubricating pigments were to be used in air, it was necessary to determine their oxidative stability at 1000°F. This was accomplished by heating the compounds to 1000°F for 4 hours and measuring the weight change and analyses of the final compound. The final step in screening

TABLE 2. CRITERIA FOR SELECTING HIGH-TEMPERATURE LUBRICATING MATERIALS

- A. Minimum melting point of 1000°F.
- B. Hardness not greater than 4.0 on Moh's scale.
- C. Insoluble in water and common organic solvents.
- D. Similar in crystal structure to presently known solid lubricants or crystal structure that appear to offer a low shear plane.

possible solid lubricants was the determination of friction characteristics. The pellet tester designed at MRI for high-temperature friction studies has shown good correlation of friction values observed in wear tests. Figure 9 shows a typical curve for coefficient of friction versus temperature as determined on the pellet tester.⁽⁹⁾ One of the major problems encountered with these high-temperature pigments has been the lack of consistently low friction at all temperature levels.

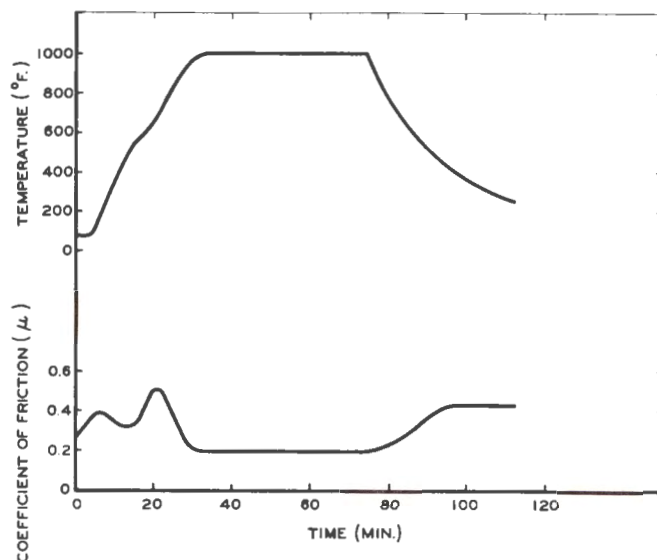


FIGURE 9 COEFFICIENT OF FRICTION OF PbS PELLETS

FIGURE 9. COEFFICIENT OF FRICTION OF PbS PELLETS

The majority of work with these films has been formulation of pigment-binder combinations which will provide long wear life. Early studies on high-temperature films led investigators to believe that binders would be a relatively minor problem due to the availability of ceramic materials with good high-temperature stability. As actual films are formulated, however, the matching of the binder to the substrate metal and compatibility with the pigment becomes a major problem. Various materials such as silicone resin, sodium silicate, sauerisen cement, boric oxide, and ceramic adhesives have been formulated with various pigments to form solid lubricants. Some of these, such as the silicone resin and sodium silicate, gave a low coefficient of friction, but showed a high rate of wear on the rub shoes. Wear life for these films is also low as shown in Table 3.⁽⁹⁾

To date the most promising film developed for operation at 1000°F has been a lead sulphide (PbS) pigment bonded with boric oxide (B_2O_3). Wear life for this film is outstanding at 1000°F as shown in Table 4, but it suffers low wear life and high friction at the lower temperatures.⁽⁹⁾ This was not anticipated since the data from the pellet test had indicated fairly low coefficient of friction and good lubricity over the temperature range 80 to 1000°F. This would indicate that the binder in such a composition is influencing lubricity and wear life to much greater extent than originally considered. This points out the need for fundamental work in this area to provide a sound understanding of the mechanisms involved. Various other pigments were tried with B_2O_3 ,

TABLE 3. WEAR LIFE OF SILICONE RESIN AND SODIUM SILICATE BONDED FILMS

Test	Lubricant	Binder	L:B (by wt)	Load, lb	Temperature, °F	Speed, rpm	Wear Life, rev	Avg. Coeff. of Friction
1	MoS ₂	Silicone	1:1	195-630	200	220	7,000	.05-.08
2	MoS ₂	Silicone	1:3	195-340	200	220	2,700	.26-.50
3	MoS ₂	Silicone	3:1	195-260	200	220	2,100	.13-.32
4	PbO	Silicone	4:1	100	500	370	2,800	.25
5	PbS	Silicone	6:1	100	1000	370	800	.27-.32
6	PbS	Silicone	9:1	100	1000	370	700	.17
7	MoS ₂	Sodium Silicate	3:1	100	750	370	2,600	.08
8	PbS	Sodium Silicate	1:1	100	500	370	400	.25-.33
9	PbS	Sodium Silicate	6:1	100	1000	370	1,600	.29
10	Bi ₂ S ₃	Sodium Silicate	6:1	100	500	370	1,300	.26
11	Bi ₂ S ₃	Sodium Silicate	15:1	100	700	370	1,600	.21
12	Bi ₂ S ₃	Sodium Silicate	6:1	100	1000	370	2,500	.23

TABLE 4. WEAR LIFE OF PbS FILMS BONDED WITH B₂O₃

Film Composition	L:B (by wt)	Temperature, °F	Load, lb	Wear Life, rev	Avg. Coeff. of Friction
PbS-B ₂ O ₃	6:1	1000	100	26,100	.06-.15
PbS-B ₂ O ₃	6:1	1000	100	35,000	.12
PbS-B ₂ O ₃	15:1	1000	100	19,300	.15
PbS-B ₂ O ₃	9:1	100	118	500	.28
PbS-B ₂ O ₃	9:1	1000	100	47,600	.08-.14
PbS-B ₂ O ₃	9:1	100	100	Failed on loading	-
PbS-B ₂ O ₃	15:1	500	100	Failed on loading	-

but the results obtained were not as good as with PbS and in many cases the coefficient of friction and wear rate were high.

The performance of the PbS-B₂O₃ films at 1000°F has prompted the current effort toward improving the wear life at room temperature. Additions of graphite to the film produced very little improvement in the film performance at any temperature. Molybdenum disulphide gave an outstanding improvement in the wear life at room temperature but had a detrimental effect at high temperature. Table 5 shows the results obtained with the addition of graphite and MoS₂ to the PbS-B₂O₃ system.⁽⁹⁾ Based on the results obtained with the PbS-MoS₂-B₂O₃ combination at room temperature, it was assumed that an optimum mixture of these components could be obtained which would improve wear life performance over the entire temperature range. Various combinations of these components are presently being tested with the percentage of MoS₂ ranging from 0.25 percent to 75 percent (by weight). The best results obtained to date have been films with a lubricant mixture of 67 percent MoS₂ and 33 percent PbS. This film gave 20,000 revolutions at 700°F and 4000 revolutions at 1000°F. These results were obtained on a wear tester of the opposed rub shoe type with a normal load of 100 lb and speed of 370 rpm. These results fall far short of the desired wear life of films for 1000°F application, but efforts along this line will be continued to improve performance over the entire temperature range. The search for other lubricating pigments and high-temperature binders continues with the hope that a combination with better wear life performance over the temperature range 80 to 1000°F may be developed.

TABLE 5. WEAR LIFE OF PbS-MoS₂ AND PbS-GRAPHITE FILMS

Film Composition	L:B (by wt)	Temperature, °F	Load, lb	Wear Life, rev	Avg. Coeff. in Friction
PbS-G-B ₂ O ₃	7:1	1000	100	200	.15-.27
PbS-G-B ₂ O ₃	7:1	100	100	1,540	.27
PbS-G-B ₂ O ₃	7:1	750	100	340	-
PbS-MoS ₂ -B ₂ O ₃ (75%) (25%)	8:1	120	100	100,000	.03-.06
PbS-MoS ₂ -B ₂ O ₃ (75%) (25%)	8:1	1000	100	2,500	.11
PbS-MoS ₂ -B ₂ O ₃	11:1	1000	100	2,000	.04-.18

Other facets of this current effort include surface pretreatment of the test races before spraying. The effects of polishing and liquid honing the races have been studied. Surface roughness of factory finished, polished, and liquid honed races has been determined from electron micrograph studies. The results showed the average roughness of the factory finished races was 3.5 microinches, that of the polished races was 2.5 microinches and that of the liquid honed races was 1.7 microinches. Preliminary results indicate substantial improvement in wear life is obtained at 700°F when a film of PbS + MoS₂ + B₂O₃ is bonded on a liquid honed substrate. Precoating the races with B₂O₃ before applying the lubricant film has been studied. Results indicate no significant effect for precoating steel races, but was found to be essential for titanium races. Films of MoS₂ + B₂O₃ on titanium failed upon loading with no precoating, but gave an average life of 45,000 revolutions at room temperature with a B₂O₃ precoating.⁽⁹⁾

An attempt to shed light on the behavior of the PbS-B₂O₃ films prompted an investigation with this combination in the pellet tester. Whereas only the pure pigment is usually tested for frictional properties, these tests were conducted on pellets of the PbS and B₂O₃ powders in the same ratio as for bonded studies. For comparison, pellets of MoS₂-B₂O₃ were also prepared. These

pellets were tested in the same manner as the pellets of PbS and MoS₂ only. The friction coefficient of the MoS₂-B₂O₃ pellets ranged from 0.06 to 0.25 as the temperature was increased from 80 to 1000°F. Some oxidation of the MoS₂ was evident and undoubtedly contributed to the observed increase in friction coefficient. The pellets of PbS-B₂O₃ exhibited a high coefficient of friction at all temperatures with the friction value increasing from 0.45 to greater than 1.0 in the temperature range from 80 to 750°F. Tests starting at 1000°F also exhibited coefficient of friction greater than 1.0. Altering the ratios of pigment to binder in the pellets of PbS-B₂O₃ indicate the coefficient of friction can be lowered. A ratio of 12 to 1 lubricant to binder shows a friction value of 0.30-0.35 while ratios of 18 to 1, 9 to 1, and 6 to 1 exhibit coefficients of friction above 0.70 at all temperatures. In contrast to this, wear life test data indicate best results for films of PbS-B₂O₃ in ratios of 6 to 1. Here again, as in the bonded studies, is the indication that the binder is playing an important role in the lubrication characteristics of these films.

Recently the high-temperature solid film program has been increased in scope to cover wear life studies of these films in vacuum environments. A vacuum wear tester has been designed and constructed at the Midwest Research Institute. The tester was designed to operate at 10⁻⁵ mm Hg with test specimen temperatures up to 1000°F, loads to 80,000 psi and speeds to 200 ft/min. Figure 10 shows the drive and loading system. A 3/4 hp DC shunt motor is used to drive the specimen. The motor is mounted vertically, its weight being supported by a self-aligning thrust bearing. Speed is continuously variable from 100 to 850 rpm with 3 percent speed regulation for 50 percent change in torque. Total revolutions are recorded for each test with a digital counter driven by a cam-operated microswitch. The reaction torque on the motor frame is transmitted to a pair of symmetrically mounted proof rings. One proof ring is equipped with a differential transformer to detect the strain. The signal of the differential transformer is amplified and recorded during each test. The output signal is calibrated directly in terms of frictional torque.

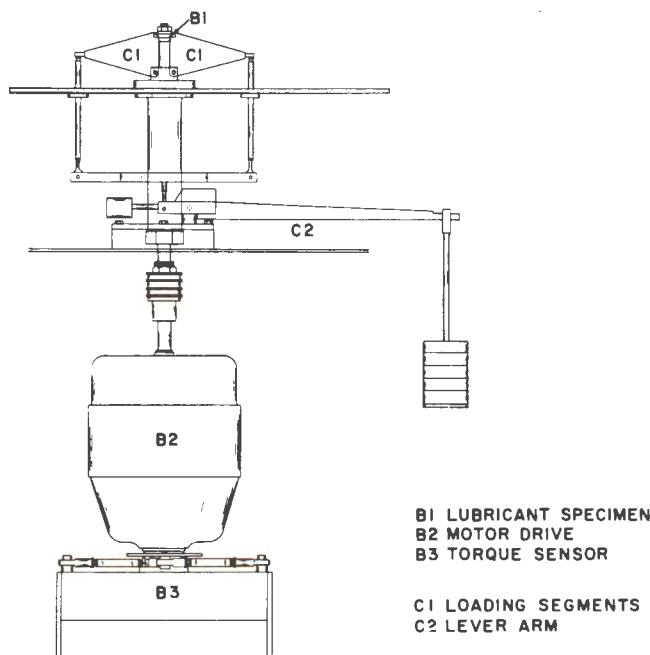


FIGURE 10. VACUUM WEAR TESTER, DRIVE AND LOADING SYSTEM

The loading system consists of a multiple lever system which applies a load on the rub shoes in contact with the test specimen. The loading lever, with a 10 to 1 mechanical advantage, transmits the load to a flat plate which drives two loading rods through O-ring seals in the base plate to the loading segments. Two self-aligning rub shoes, 180° apart, are held in contact with the test specimen. The system is designed to transmit loads of 650 lbs to each rub shoe, for this vacuum equipment.

Four films with considerable wear life data in air were chosen for the initial comparative wear life studies in vacuum.⁽⁹⁾ Tests were conducted at approximately 100 rpm and at pressures

near 5×10^{-6} mm Hg. Table 6 shows the test conditions and wear life results.⁽⁹⁾ The first three lubricants show wear lives that are one-tenth the average values in air. These are graphite films and the drastic reduction in wear life of the film may be due to the behavior of graphite in an atmosphere on which the partial pressure of water vapor is insufficient. The wear lives of MoS₂ bonded with B₂O₃ are comparable to those obtained in air. Several other tests conducted with the MoS₂-B₂O₃ film have given similar results.

TABLE 6. WEAR LIFE OF SOLID FILMS IN VACUUM

Lubricant	Temperature, °F	Load, lb	Speed, rpm	Wear Life, rev	Avg. Coeff. of Friction	Avg. Press., mm Hg
Lub E	150	120	100	25,600	.05	5×10^{-6}
Lub E	150	215	100	240	.04	3×10^{-6}
Lub A	150	215	100	540	-	5×10^{-6}
Lub A	150	70	100	36,300	.08	5×10^{-6}
M ₇	375	215	100	1,700	.04	5×10^{-6}
M ₇	375	215	100	3,200	.06	3×10^{-6}
MoS ₂ -B ₂ O ₃ (2:1)	375	215	100	20,700	.04	5×10^{-6}
MoS ₂ -B ₂ O ₃ (2:1)	575	215	100	900	-	8×10^{-6}
MoS ₂ -B ₂ O ₃ (2:1)	760	215	100	700	.04	1×10^{-6}
PbS-B ₂ O ₃ (6:1)	760	120	100	800	0	1×10^{-6}

The three component systems of PbS, MoS₂, and B₂O₃ have been tested in vacuum with varying results. The latest of these films have been prepared in an effort to maintain a fixed ratio of the number of lubricant particles to binder weight. Various combinations were tested at 400, 700 and 1000°F. Films with the greatest ratio of either PbS or MoS₂ yielded low wear lives at all temperatures. The best wear life results to date in vacuum (10^{-5} mm Hg) were obtained with the film ratio of 13:13:2. Two thousand revolutions were obtained at 1000°F, 20,000 at 700°F and over 200,000 at 400°F.

A program conducted at WADD on the PbS-B₂O₃ film has been completed. The program was designed statistically to provide data at two levels of temperature, speed, and load. These tests were conducted on the Hohman A-6 tester. Test conditions were temperatures of 800 and 1000°F, speeds of 200 and 600 rpm, and loads of 200 and 600 lbs. Wear life results were similar to those found for the resin-bonded films. The analysis of the data is near completion and will be published in the near future.

FUTURE REQUIREMENTS AND RESEARCH FOR EXTREMELY HIGH TEMPERATURE

The requirements for lubrication at 1000°F was short lived and has now been pushed up into the range of 1500 to 2000°F. At these temperatures the solid films we known now will no longer operate. Pigments as well as bonding agents that are stable in this temperature range will have to be developed. Present substrate metals will have passed their limit in strength and oxidative stability. New exotic bearing materials will have to be developed and made compatible with lubricating systems. New concepts in lubrication will be derived. Perhaps the so-called dry lubrication methods will prove to be effective at these temperatures.

The Air Force programs in high-temperature lubrication are being reoriented to cope with these increased requirements. A vacuum wear tester, very similar to the one described earlier, is being constructed currently with temperature capabilities above 1500°F. Compounds with melting points and thermal stability above this temperature are being screened for possible lubricating pigments. New bonding techniques are being investigated. A program recently started at Southwest Research Institute is investigating lubrication and bearing design for operation at 2000°F. This marks the first attempt in this area to design a bearing and develop a lubricating technique at the same time. It is felt much greater success will be attained with this concept than with trying to lubricate with solid films a bearing designed for grease or oil. Some of the compounds of beryllium, columbium and other exotic metals are being investigated as possible bearing materials. Equipment has been designed to provide friction and wear studies at 2000°F.

More basic research programs are being conducted to provide a better understanding of the mechanisms involved in friction and wear. Studies at high sliding speeds between hard and soft, soluble and insoluble metals, for example, are being conducted. The phenomena associated with interfacial temperatures and methods for determination of these temperatures are being investigated. Efforts to delve into the heart of some crystalline compounds, to discover their lubricating secrets, determine bond energies, etc., are already underway.

Not forgetting the problems facing us today with strangely behaving binders, studies will be initiated to investigate bonding mechanisms, reactions within the composition, thermal expansion coefficients, etc. Answers to these questions are needed in order to understand and develop more efficient bonding agents. High-temperature inorganic ceramic adhesives will be investigated as possible binders; modification of these adhesives with metal salts, surface pretreatment, etc., will be attempted to improve properties such as ductility, thermal expansion, greater bond strengths, etc.

In summary, these programs point toward fundamental mechanisms, a greater understanding of the problems at hand, and vigorous efforts to keep abreast of the ever increasing requirements of both current and future weapon systems.

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