

94-14990 289

ASME PUBLICATION



10 PER COPY

50 C TO ASME MEMBERS

The Society shall not be responsible for statements or opinions advanced in papers or in discussions of members of the Society or of its Divisions or Sections, or printed in its publications.

Discussion is printed only if the paper is published in an ASME journal.

Released for general publication upon presentation.

94-14990



128

BEST AVAILABLE COPY

Solid Films - Lubricants for Extreme Environments

C. F. MERRILL

Project Officer,
Fluids and Films Section,
Material Laboratory,
Wright Air Development
Division USAF, Dayton, Ohio.

ROBERT J. BENZING

Chief, Fluids and Films Section,
Material Laboratory,
Wright Air Development
Division USAF, Dayton, Ohio.

DTIC
ELECTE

MAY 26 1994

S F D
COPY 1

LIBRARY COPY

The function of this paper is to review the area of solid film lubricants, their present use, and requirements for future developments. Particular emphasis will be placed on application in military programs. As will be seen later in the paper, solid films are currently in wide use in military vehicles with most applications being found in slow sliding bearings at loads from 2000 to 80,000 psi. Present films have long term capabilities at temperatures to around 400 to 600 deg F depending upon the specific application. The development efforts to be reviewed are being carried out in the 1000 to 2000 deg F temperature range. It is in this particular range that solid films offer considerable promise above and beyond the use of such standard lubricants as greases and oils.

This document has been approved
for public release and sale; its
distribution is unlimited.

95866

DTIC QUALITY INSPECTED 8

For presentation at the Petroleum Mechanical Engineering Conference, New Orleans, La., September 18-21, 1960, of The American Society of Mechanical Engineers. Manuscript received at ASME Headquarters, June 16, 1960.

Written discussion on this paper will be accepted up to October 19, 1960.

Copies will be available until July 1, 1961.

94

078

1

Solid Films - Lubricants for Extreme Environments

C. F. MERRILL

ROBERT J. BENZING

At the outset it is important to understand what is meant by the term "solid film lubricants." We use this term to designate a series of lubricating materials consisting of a friction-and-wear-reducing pigment, such as graphite or molybdenum disulphide, bonded to the bearing surface with some type of adhesive. Current films use such adhesives as the epoxy resins while work in the 1000 F range is considering ceramic-type materials. Another term generally used is that of "dry-film lubricants." This is used in many cases as synonymous with solid films. In our work, however, we use this term to refer to materials which chemically react with the metal surface or are plated on to provide wear resistance. These materials are now being studied as possible high-temperature materials. These particular types of materials cover an area of wide scope of their own and it is not intended to review them in this paper.

As can be deduced by the title of the paper, solid films are generally considered for use in areas of extreme environments. Such environments include extremely high temperatures as we have seen, temperatures in the cryogenic region, areas of high-intensity nuclear radiation and hard vacuum applications. That is, they are called on to perform in applications where greases and oils rapidly degenerate and are useless. This is mainly due to the fact that design engineers first think of greases and oils as the lubricating media because of their past association and only discover solid films when these other materials will not suffice. This does not mean, however, that solid films will not work in the less severe applications and, as we will see, are actually finding their way into such areas. Solid films have one particular attribute which makes them attractive for any application in addition to their stability in extreme environmental conditions. It is the property or concept of lubrication for the life of the system with no external lubrication network. Once put on a bearing surface, solid films are expected to last the life of the component or until normal bearing maintenance and removal periods. The fact that they are thus applied also results in considerable weight and money savings by eliminating the complex lubrication system and/or maintenance encountered with other materials.

BRIEF HISTORY AND REVIEW

Early work in the area of solid film lubricants resulted from the known behavior of low friction of graphite, MoS_2 and other related materials. Godfrey and Bisson (2)¹ conducted early investigations on bonding of a friction-reducing solid, MoS_2 , to various metal surfaces including steel, aluminum brass and stainless steel. Other investigators (3, 4, 5) have studied the use of polytetrafluoroethylene (PTFE) as a friction-reducing coating. It has good characteristics from a friction standpoint but is limited to light loads and temperatures in the order of 600 F.

A wide scope of literature can be found on the early work of graphite and MoS_2 bonded to surfaces. Results on various commercial films are presented by Hart and Rubin (17). However, these two materials are limited to the lower temperatures due to their chemical and/or physical limitations. Early work on graphite in electrical equipment demonstrated the need for an adsorbed water layer to produce low friction. An excellent summary of the problem is given by Atkins and Griffiths (6). Bisson, Johnson, and Anderson (7) present data at 1000 F in which they show low friction with graphite at very elevated temperature, however, and draw the hypothesis that in addition to having low friction with adsorbed water on the graphite it is possible to produce the same results with an oxide film on the lubricated surfaces. This oxide film is felt to influence the adherence of the graphite to the surface and result in low friction. MoS_2 , in turn, has been shown to oxidize at around 750 F by Godfrey and Nelson (8). This limits its long-term use at extremely high temperature due to the high friction of the oxidation product.

High-temperature work has been conducted on a wide variety of lubricating pigments for use at 1000 F and above (9, 10, 11). These would substitute for the graphite and MoS_2 . Such pigments include FeO , CdI_2 , CdCl_2 , PbS and NiCl_2 . Actual film formulations with ceramic and other high-temperature binders have been reported in

¹ Numbers in parentheses designate References at the end of the paper.

the literature (10, 12) and promising results given. As we will see, solid films show exceptional performance at 1000 F.

PRESENT CAPABILITIES

Dry-film lubricants on the market today are composed of a pigment or friction-reducing mitigant, a binder which is in most cases an organic adhesive, and a thinning solvent to give the proper consistency for spray application or dip coating. This mixture is applied to a metallic bearing surface which has been subjected to some form of pretreatment to increase the adhesive bond between the metal and the film. The relative merits of one film over that of another rest in the differences in the pigments selected, the binders used, the pigment-to-binder ratio, and the compatibility of these two with each other in a cured composition. In the majority of cases, the constituents of these films are identical or very nearly so. The differences between successful films and poor films are usually due to trial-and-error selection of materials. Such formulations eventually result in chemical constituents and compositions which give satisfactory performance. Such Edisonian type research is required because of a lack of fundamental understanding of the mechanisms involved.

Binders fall into the two categories of organic resins and inorganic adhesives. The organic materials are by and large phenolic and epoxy resins with the majority being epoxy compositions. Some silicone resins have been used but not with a great deal of success. The inorganic binders available are metal matrix compositions, glasses, and ceramics. The compositions of the metal matrix and ceramic binders used commercially are proprietary to the individual companies and, therefore, will not be specifically defined. Work which has been done by the Air Force in ceramic binders will be discussed later in the research portion of this paper.

Pigments for the low-temperature solid films are almost entirely molybdenum disulfide and graphite. Some films incorporate either one or the other of these pigments in the film while others use a mixture of the two. In the cases of the mixture, it has been the practice to use approximately 80 to 90 per cent molybdenum disulfide and the remainder graphite. The actual influence of the presence of this small percentage of graphite has never been explained scientifically; however, bench evaluation and service application have demonstrated its advantage. Other friction-reducing materials have been considered; however, relatively few are on the market. Ex-

tensive evaluation of potential pigments has revealed promise for new materials, and hope for extreme environments (10). These will be discussed further under research for future requirements.

In general, it may be stated safely that the films composed of the materials just discussed will provide satisfactory lubrication from room temperature to 400 to 500 F for relatively heavily loaded, slowly sliding bearings and for reasonably rapid speed, lightly loaded parts. The properties of this type film as an antisieze materials are also outstanding.

These are the characteristics of present dry film lubricants. What then is the prerequisite for the metal surface to which the lubricant 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 (13, 17). Since this has such a tremendous influence on performance it is wise to consider the various possibilities and be able to employ them successfully. Commonly used surface treatments for ferrous metals include iron and manganese phosphating, vapor and grit blasting. Study of methods has shown that phosphate treatment has the largest influence in increasing wear life. Unfortunately this method of pretreatment is limited to metallic substrates of iron and steel. Other metals use other less successful treatments. The phosphate pretreatment creates a nonmetallic coating consisting chiefly of iron and manganese phosphates deposited in a crystalline form on the substrate metal. The grit blast and vapor blast are purely mechanical workings of the metal surface to create a roughened area allowing the binder material more surface area contact to increase adhesion. In general, surfaces should be roughened to a surface greater than 15 RMS and a more acceptable figure would be in the neighborhood of 35 RMS. In grit blasting 120-mesh grit is usually employed.

Unfortunately, the usage of the phosphating procedure is not only limited in materials to which it is applicable, but also in its method of application. Probably the largest influence on phosphating iron and steel surfaces is that of bath and solution control (13). Temperature of the bath is all important in assuring proper crystal deposition and must be controlled within 5 to 6 deg of the determined optimum temperature. Work conducted by one supplier has shown that for most iron materials a bath with an acid strength of 7 to 8 points at a temperature of 205 F and immersion time of 15 min. produces the best crystal deposit. For this treatment it is

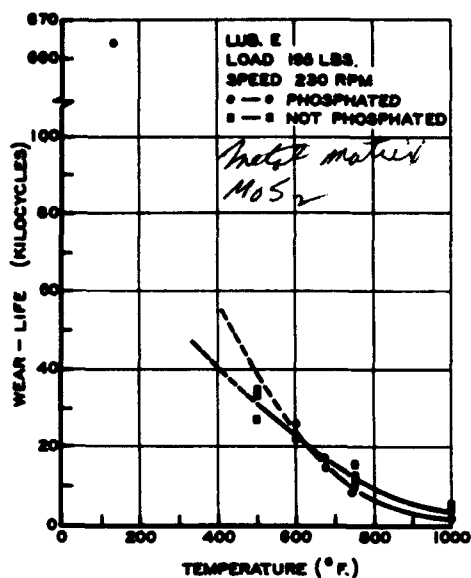


Fig. 1 Effect of temperature on wear life of phosphated and non-phosphated bearing surface

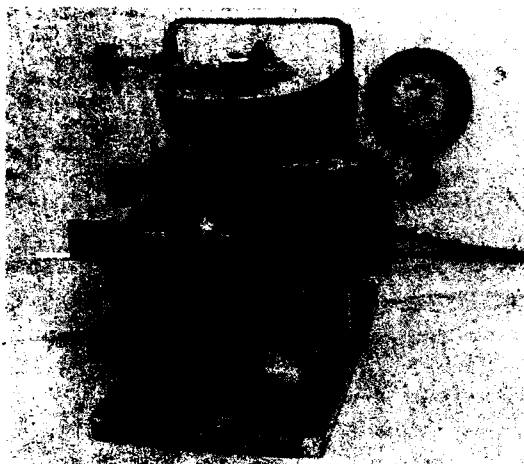


Fig. 2 Falex lubricant tester

necessary to have a uniform small crystal growth on the metal which the aforementioned conditions will produce satisfactorily. It also should be pointed out that the conditions of this treatment are dependent upon the material used. If a different iron alloy is used the optimum conditions may vary considerably. Since the mechanism of the kinetics of this pretreatment procedure have never been defined chemically, it is impossible to determine the proper bath conditions beforehand. The proper bath is determined from experience of a trial-and-error nature. Many users of solid-film lubricants are not aware of

the great influence which the conditions of the bath play on the treated surface and, as a result, often employ lax measures in controlling the conditions. This results in poor surfaces for the lubricant.

Another major drawback in the phosphate pretreatment is its temperature limitation. Although some films may have materials incorporated in them which are stable above 600 F their optimum performance is limited to this temperature if they have a phosphate pretreatment. Data typical of this reduction in endurance life are shown in Fig. 1 (10). As can be seen from the figure, the crossover point for phosphate and nonphosphate 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, this film with a phosphate pretreatment have given satisfactory results above 100,000 cycles under the same test conditions. Noting the difference between trends substantiates our earlier statement that phosphating increases wear life many fold. The criterion for failure in this test is friction increase to a value of 0.1 or better. It appears, therefore, that since phosphating is unsatisfactory above 600 F dependence will have to be placed upon mechanical working of the surface as with grit or vapor blast or development of a better high-temperature pretreatment.

Let us now shift our attention to the effects of various environments on the solid-film lubricant itself. A great deal of work has been conducted on commercial films and also on research compositions. Before reporting on this it is necessary to have a common basic understanding of some of the evaluation tests used for solid-film lubricants so that the results are more easily understood. Very few laboratories today employ a test apparatus which evaluates a particular application such as a bearing, an actuator, or a hinge. Most of the evaluations are performed with test devices which attempt to simulate the various conditions to be encountered. Such techniques have been developed for the simplicity of the test specimen and the rapidity with which tests may be run. These machines evaluate the relative merits of one film to another under the desired test conditions and report a parameter such as endurance time to failure. A determination of a certain number of hours wear life for a particular test on a film does not mean that the film will perform in

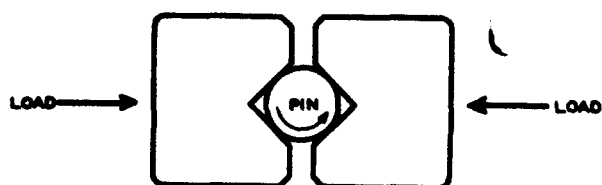


Fig. 3 Principle of loading and operation Falex lubricant tester

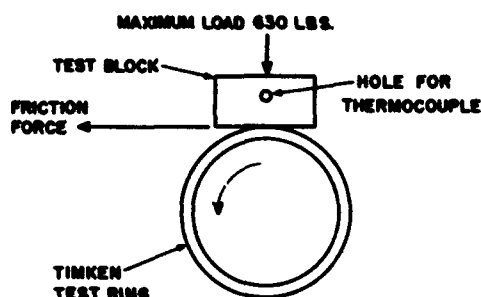


Fig. 4 Principle of loading and operation - block and shoe wear tester

the same manner in an actual application. If it has shown, however, that it is better than another film under the same conditions, this relative relationship will probably hold in the actual usage. In other words, the equipment used does not give a 1 to 1 correlation; but it does provide for screening of various films. Probably the most widely used piece of apparatus in this category is the Falex lubricant tester.

The Falex employs a coated test pin 1/4-in. diam rotating between two coated V-blocks loaded to the desired test conditions through a jaw-lever-arm system. The standard Falex is shown in Fig. 2. A schematic of the test area is shown in Fig. 3. Tests on this machine may vary from several minutes to 2 or 3 hr, depending upon the test conditions and the film studied. The other type of test apparatus commonly used is a block sliding on a rotating cup for which there are many variations. The basic tester employs an outer race of a Timken bearing as a wear surface to which the solid-film lubricant has been applied. This is rotated unidirectionally at a constant speed in contact with a rub shoe which is affixed to a lever-loading apparatus and loaded to the desired level. Several investigators have modified this type tester. These modifications include changes in the loading systems and the torque instrumentation to provide greater reproducibility in testing conditions, but the basic movement is essentially the same.

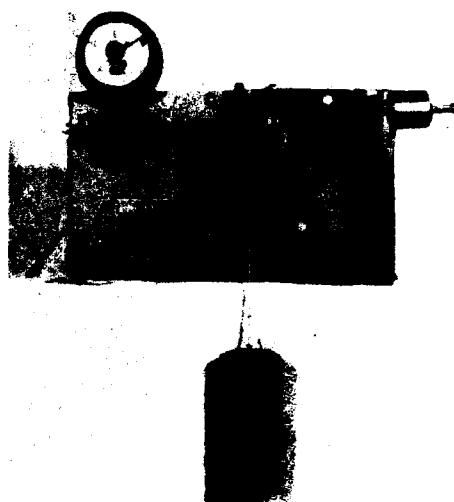


Fig. 5 Alpha lubricant tester

Fig. 4 shows a schematic of the test area with the rub shoe and rotating specimen. Fig. 5 shows the Alpha lubricant tester, one modification of the basic test. Neither the standard Falex nor the Alpha tester, as shown, is 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 employed on the majority of the new testers, and the addition of another rub shoe located diametrically opposite is also used in some designs. Use of two rub shoes diametrically opposed eliminates the problem of shaft deflection under high loads and seems to provide greater reproducibility. In addition, the introduction of another wear surface reduces the time necessary for testing.

In observing data received from a particular test apparatus, care should be taken to notice the use of either single or double rub shoes since the effect of two shoes will decrease the number of hours run yet the feet traveled by the wear surface may be quite comparable. These new testers have been developed at several laboratories. All are capable of operation at temperatures to 1000 F and some are capable of operation in such unusual environments as hard vacuums (10^{-6} mm Hg or better), contaminating vapors, and cryogenic temperatures. Fig. 6 shows a Hohman A-6 tester. Its capabilities are 1500 F temperature, light and heavy loads and various speeds. In addition, the wear surface is housed in an environmental chamber capable of handling unusual corrosive contaminants as well as vacuum.

The Air Force has been interested in per-

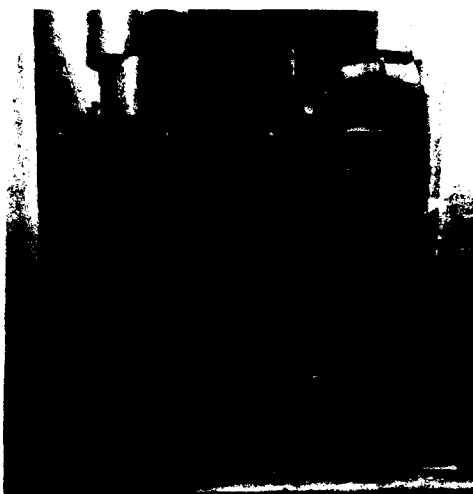


Fig. 6 Hohman A-6 lubricant tester

formance capabilities of dry-film lubricants at temperatures from the cryogenic level to 1000 F or better and in such environments as nuclear radiation. More recently, performance under hard vacuum has become of interest and will be discussed further in the research portion of this paper.

As was mentioned earlier, Midwest Research Institute was contracted by the Air Force to develop bonded high temperature solid lubricants. The initial work studied the capabilities of presently available films. Parameters included in this study were environmental temperature, bearing load, substrate hardness, film thickness, relative linear surface speed, lubricant material, film binder, lubricant-to-binder ratio type of motion (unidirectional or oscillatory) and the geometry of the rub shoe surface; i.e., whether it conformed to the test surface or presented essentially line contact. Owing to the number of parameters to be investigated and to the number of tests necessary to obtain good confidence in a piece of information a statistically designed experiment was set up. This allowed for evaluation of a lubricant with a minimum number of tests. Two levels of testing were chosen within each parameter. The criteria for choice of each level were:

Values in the range of interest

Values expected to yield significant results.

Values within the practical and convenient limits of experimental equipment.

The equipment used in this testing program was of the block-and-shoe type employing two wear shoe surfaces. The final statistical approach selected was an experimental setup em-

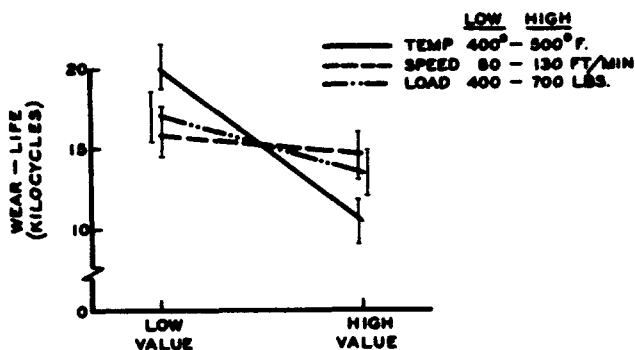


Fig. 7. Statistical analysis of wear life of solid films

playing 1/2 replicate of a 25 factorial design. The results of this evaluation showed that the parameters having the predominant effect on the wear life are temperature and load. Other factors had an effect on the wear life but not nearly as great as the two just mentioned. The most important concern to the Air Force and one which turned out to be a predominant factor was the decrease in wear life as temperature increased. The two temperature levels selected for this testing were 400 and 550 F. The 550 F temperature was chosen as it approaches the upper limit of present films. The 150 F temperature spread was considered adequate to show the temperature effect. Fig. 7 illustrates the effects of temperature and load as determined in the factorial experiments. As may be observed, there was a drastic change in wear life when going from low temperature, high loads and high speeds to the high temperatures and low loads and low speeds. The need for research in improving the wear life of solid-film lubricants, as environmental temperatures increased, became evident.

A good deal of solid-film evaluation has been performed at Wright Air Development Division. To date, the greater majority of the work has been room-temperature evaluation on the Alpha tester described earlier. Results from 3 years of evaluation with this apparatus have shown that present solid-film lubricants should be capable of an average of 70 hr continuous operation. Test conditions have been 630-lb normal loads, unidirectional motion of 72 rpm and ambient room environments.

Another program conducted at WADD was the evaluation of solid lubricants at extremely low temperatures. For this study a standard Falex tester was modified to allow submersion of the test area in liquid nitrogen (bp -196 C). Fig. 8 shows the modifications of this tester. Instrumentation included a recording wattmeter to mon-

Table 1 Performance of Solid Films in Liquid Nitrogen

Wear Life in Minutes in Falex Tester at 1000 lb Load					
	Run 1	Run 2	Run 3	Run 4	Avg Loss in Life
Film A					
Room Temperature	202	202	203	215	88%
Liquid N ₂	16	14	42	24	
Film B					
Room Temperature	446	446	425	370	96%
Liquid N ₂	10	19	27	11	
Film C					
Room Temperature	380	250	300	247	97%
Liquid N ₂	8	9	11	13	
Film D					
Room Temperature	473	407	436	433	96%
Liquid N ₂	27	18	20	15	

itor torque and a temperature recorder to indicate specimen temperature. Evaluation of films from seven commercial vendors was conducted on this test apparatus and results for four of these films are shown in Table 1. Results showed that a 85 to 95 per cent decrease in the 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 disulfide, graphite, combinations of these two, and 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. Apparently the properties of PTFE which cause it to fail at higher loads do not affect its lubrication at the cryogenic temperatures and light loads. One major property which causes failure at high temperature but is relatively unimportant under cryogenic is PTFE's poor thermal conductivity. In addition to this, the mechanical properties of PTFE improve considerably at the cryogenic temperatures.

Another environmental effect receiving a great deal of attention is that of nuclear radiation. Environments existing in radiation belts surrounding the earth and those to be encountered in proposed nuclear power systems create a concern for the operator of materials in systems encountering these environments. To study the ef-



Fig. 8 Falex lubricant tester converted for cryogenic testing

fects of these environments on available solid-film lubricants, programs were conducted at WADD and at Midwest Research Institute.

The program conducted at WADD was on commercially prepared specimens using the Falex wear tester and a block-and-shoe-wear machine. Zero level radiation tests were run and compared in wear life to runs made at increasing radiation levels. In addition to the wear-life studies, radiation effects on corrosion resistance, fluid resistance and thermal stability were performed. Corrosion tests were in accordance with established military corrosion tests in salt spray. Fluid tests were a check on adherence of the film after exposure to radiation when left in common aircraft fluids for a specified time. Thermal stability tests were a resistance to 500 F and -65 F. Levels of radiation of gamma exposures from 8.71×10^9 through 2.61×10^{11} ergs per gram carbon and neutron exposures from 1×10^{15} to 3×10^{16} nvt were studied. Radiation of samples was performed at the Materials Test Reactor, Idaho Falls, Idaho. After irradiation, samples were returned to WADD for evaluation. Tables 2 and 3 show the wear-life results. It was observed that radiation in general had very little effect either on the wear life of the film or on the corrosion resistance, fluid resistance, and thermal stability.

The work conducted at Midwest Research Institute was similar in nature to that at WADD; however, all tests for wear life were run on the MRI test apparatus which is a block-and-shoe-type machine employing two rub shoes diametrically opposed. No corrosion, fluid, or thermal tests were conducted by Midwest. Again

Table 2 Fatigue Wear Life for Neutron Irradiated Solid Film Coated Pins and V-Blocks

Neutron Dosage (nvt fast)	Control		1×10^{15}		3×10^{15}		1×10^{16}		3×10^{16}	
	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)
A	55° 81 24 (53)	2.7 4.1 ---	120 154 (137)	4.4 6.0	58° 38° (48)	3.2 5.0	89 179 (134)	4.6 2.8	52° 33° (43)	3.7 4.6
B	101° 84	17.4 11.7	136° 119° (128)	9.8 6.8	118° 127° (123)	11.8 10.2	99° 149° (124)	10.5 9.3	194 153 (174)	16.9 46.0
C	371 285 538	6.2 8.9 7.7	394 495 (445)	28.7 39.0	625 370 (513)	19.9 14.3	1045 894 (970)	16.0 23.1	656 346 (501)	26.7 15.5
D	Pins lost		41° 57° (49)	24.5 68.5	17° 36° (27)	---- ----	41° 68° (55)	7.8 11.8	20° 59° (40)	---- 10.2
G	409 443 (426)	19.9 17.7	165° 152° (159)	17.0 8.0	216° 271° (244)	7.4 7.4	304° 339° (322)	7.7 6.0	245° 360° (273)	7.1 7.0
H	111 100 (106)	10.8 10.7	69° 28° (49)	3.6 ----	51° 116° (84)	2.7 3.7	86 72 (79)	2.6 3.7	9 5 (7)	---- ----

* 355 lb. load (all other data for 250 lb. load).

Note: Values in parenthesis () represents average wear life.

tests were run in both gamma and neutron exposure and at the same dosage levels. In general, as far as radiation resistance is concerned it can be stated that films which possessed good corrosion resistance, fluid resistance, and thermal stability prior to irradiation are not seriously changed by these environments. The same is true for the wear life, in fact in some cases it was observed that radiation seemed to improve the performance. The radiation levels attained in this work have been found to be of sufficient strength to change completely the physical properties of our best oils and greases turning both to solid rubber-like masses. Presently available solid-film lubricants are, therefore, satisfactory in performance to approximately 500 F under reasonably heavy loads and slow speeds and the presence of gamma and neutron radiation environments.

PRESENT RESEARCH TO MEET FUTURE REQUIREMENTS

We have seen the present capabilities of solid-film lubricants and now let us turn to some of the future requirements and problems. Since standard lubricating techniques and present solid-film lubricants will not meet the demands of future 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; hence the demand for solid-film lubricating systems employing materials stable under these environments is essential.

Because of this dependence upon solid-film lubricants, many new applications formerly employing greases and oils have arisen.

The types of motion and the actual hardware for which these films are intended will include sliding bearings such as plain spherical, rod-end, and ball sockets, ball and roller bearings both large and small, ball and screw actuators, and hinge pins to name a few. These applications, as can be seen, include just about all forms of motion encountered by lubricants. The need for solid films in each one of these applications has already been witnessed and will increase. In the past, solid films have been mainly employed in sliding-type applications but the foregoing include rolling motion also. For example, we have had requests for solid films to be used at 1200 F, in small accessory bearings at 15,000 rpm and light loads. As we will see, solid films have been evaluated to some extent in similar applications but at lower temperatures.

The environmental conditions to which any one or all of these bearing systems will be subjected include hard vacuums as may be encountered in space, nuclear irradiation at both high and low flux levels for varying periods of time and extremes in temperature from those associated with cryogenic applications to those in excess of 1500 F. Atmospheres surrounding the bearings may consist of inert gases, propellant exhaust gases and oxidizing conditions. It is improbable that one application of solid lubricants will encounter all of the foregoing condi-

Table 3 Fatex wear Life for Gamma Irradiated Solin Film Coated Pins and V-blocks

Gamma Dosage (ergs/gm C)	Control		8.71×10^9		2.61×10^{10}		8.71×10^{10}		2.61×10^{11}	
Coating	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)	Wear Life (Min.)	Total wt. Loss (mg)
A	55° 81 24 (53)	2.7 4.1 ----	47° 97 130 (114)	5.0 6.6 5.0	26° 5 ----	----	33° 42 ----	4.8 6.5 ----	4° 71 102 (87)	---- 3.8 4.7
B	101° 84	17.4 11.7	124° 113	10.4 10.9	90° 123 93 (108)	4.9 14.0 12.1	43 39 (41)	10.8 17.6 ----	17 113 185 (105)	---- 10.3 15.2
C	371 285 538 (399)	6.2 8.9 7.7	190 153 383 (242)	5.9 2.9 8.2	189 255 (222)	8.2 7.2 ----	172 479 (326)	5.3 9.7 ----	513 189 (351)	9.6 7.4 ----
D	Pins lost		58 9 (34)	----	106 7 (57)	2.9 ----	74 116 69 (86)	2.6 3.1 3.0	65 111 (88)	3.8 3.2 ----
E	25 43 (34)	---- 9.8	16 9 (13)	----	20 11 14 (15)	----	52 11 7 (23)	6.9 ---- ----	101 138 (120)	10.5 8.6 ----
F	3 4	---- ----	3 3	---- ----	2 1	---- ----	2 2	---- ----	3 3	---- ----
G	409 443 (426)	19.9 17.7	504 349 (427)	27.6 18.1	423 528 (476)	37.4 32.7	319 699 (506)	17.9 33.6	475 521 (498)	19.8 18.3
H	111 100 (106)	10.8 10.7	39 84 (57)	11.2 9.4	63 92 (79)	17.0 5.0	32 89 (57)	24.7 8.4	88 74 (77)	8.1 7.4

* 355 lb. load (all other data for 250 lb. load).
Note: Values in parenthesis () represent average wear life.

tions and therefore solid films quite likely will be tailored to each specific application.

In the area of sliding applications, effort has been mainly 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 areas mentioned.

NACA conducted some of the initial work on materials usable to 1000 F. The most promising of their films consists of PbO bonded to the surface with silicate of soda (11). This film has shown low friction values at 1000 F but increases in friction at lower temperatures. Testing by NASA on this film consists mainly of a simple spherical rider rubbing on a flat surface. Further evaluation of this type of film in a block-and-shoe apparatus indicates lower relative wear life when compared to other high-temperature films.

Midwest Research Institute has conducted a survey of over 22,000 compounds (15) and has found approximately 1500 materials with melting points over 1000 F. Their initial survey was then used for selection of about 60 solids as possible pigments (10). These materials were selected using the following criteria:

Minimum melting point 1000 F.

Hardness must be less than 4.0 on Mohs scale.

Insoluble in water and common organic solvents.

Similar crystal structure to presently known solid lubricants or crystal structures that appear to offer a low shear plane.

Friction tests shown in Table 4 on these materials again indicate the one major problem encountered in high-temperature pigments; that is, the lack of consistently low friction at all temperature levels. The most promising film developed to date by MRI has been a PbS pigment bonded with B_2O_3 . Table 5 compares the results of this film to a PbO- B_2O_3 type film and a commercial film. However, it also has reduced wear life and high friction at low temperatures as shown in the table.

Close examination of these programs and other high-temperature research tends to point out a critical problem in the area of binders. Early studies on high-temperature films led investigators to believe that binders would be a relatively minor problem because of the availability of ceramic materials with good high-temperature stability. As actual films are formulated, however, the matching of the binder to the substrate and compatibility with the pigment become major problems. No fundamental work has been done in this area to provide a sound understanding of the mechanism involved.

As mentioned before, surface pretreatments

Table 4 Friction Data on Various Solids
as Possible 1000 F Film Pigments^a

Compound	Coefficient of Friction		
	80 F	500 F	1000 F
PbS	0.08	0.47	0.21
CdS	0.58 and 1	0.84	0.55
AlPO ₄	1.33	0.31	0.31 - 0.37
Sb ₂ S ₃	0.38	0.21-0.49	0.49
AgI	1	1	1

^a Data from Midwest Research Institute

have a remarkable effect on improvement of wear life. At high temperatures the only known methods are mechanical roughening of the surface and undercoats of various nonpigmented adhesives. Until final solid films and bearing materials are selected, however, little work can be conducted on pretreatment development. This is due to the intimate interaction of the film, bearing material, and pretreatment process. At this time we contemplate use of such bearing materials as the tool steels, Inconel X, carbides, and other high-temperature alloys which are becoming available commercially.

Lubrication under conditions of rolling friction is a relatively new application for solid-film lubricants. Rolling applications include such things as antifriction bearings and ball-screw actuators. Even in such applications as a ball bearing there is a considerable component of sliding motion as between the ball cage and the ball itself. The sliding speeds in ball bearings are well above normal solid-film uses. In a 204 bearing operating at 50,000 rpm (10^6 DN) the sliding speed is 13,300 fpm. From another aspect, however, loads in antifriction bearings are well below those encountered in normal solid-film use.

For many years solid films were not considered for antifriction bearings. However, within the past year Devine and Lamson (16) have published some interesting work on the use of solid films in 204 bearings operating at 10,000 rpm. Table 6 summarizes the extent of their work. Although life of the film was relatively short, the bearing mechanical performance was the limiting factor as opposed to temperature. Such results indicate future effort in this area holds great promise. Currently WADD is contemplating a study of rolling and sliding friction with solid films using the Pratt and Whitney one-

Table 5 Wear Life on Candidate 1000 deg F Solid Films
370 RPM - Dual Shoe Tests

Film	L/B* Ratio	Load (lb)	Temp (°F)	Wear Life (Cycles)**
PbS-SiO ₂	6/1	100	1000	30,500
	1/1	100	Room Temp	500 and failed on loading
	6/1	200	1000	29,000
PbO-SiO ₂	6/1	200	500	Failed on loading
	6/1	---	1000	38,000***
Commercial Film	---	200	400	33
	---	---	700	108

* Pigment to resin ratio.

** wear life determined by increase in friction.

***No friction change but drastic wear which would reduce actual wear life from this value.

Table 6 Performance in a 204 Bearing at 350 F

RPM	Hours to failure
1250	240
3500	42
10,000	29

ball fatigue tester. With this apparatus we will attempt to simulate the conditions existing at the sliding and rolling surface of an antifriction bearing. It is hoped that results of this program will define the limitations and indicate future development requirements of solid films in such applications as well as point the way to required bearing-design concepts.

CONCLUSIONS

Wear evaluation at high temperature has been reviewed briefly. In addition, many of the aircraft companies have conducted film evaluations on a wide range of actual bearing testers. In general, these data have not been reported in the open literature.

To date little work has been conducted on solid films in radiation or vacuum environments. However, extreme interest has been generated in these areas owing to the space and nuclear-powered aircraft programs. Initial work conducted by MRI in vacuum and to date unpublished has revealed a deteriorating effect in hard vacuum (10^{-6} mm Hg) on wear life. However, solid films are expected to far outperform liquid and grease-like materials. Due to their high vapor pressures, standard lubricants are decidedly limited for applications under high vacuum conditions.

HEADQUARTERS

Wright Air Development Division

AIR RESEARCH AND DEVELOPMENT COMMAND

UNITED STATES AIR FORCE

WRIGHT-PATTERSON AIR FORCE BASE, OHIO

REPLY TO
ATTN. OF:

WWRCNL-1 (Mr. Benzing)

31 MAR 1961

SUBJECT:

Paper, "Solid Films - Lubricants for Extreme Environments"

to National Aeronautics and Space Administration
Lewis Research Center
ATTN: George Mandel
21000 Brookpark Road
Cleveland 35, Ohio

Attached is subject paper by C.F. Merrill and R.J. Benzing as requested in
your letter dated 22 March 1961.

FOR THE COMMANDER



R. J. BENZING, Chief
Fluids and Films Section
Fluids and Lubricants Branch
Nonmetallic Materials Laboratory
Materials Laboratory

1 Atch
Paper by Merrill & Benzing

REFERENCES

- 1 Proceedings of the Air Force - Navy - Industry Lubricants Conference, WADC Technical Report 57-93, November 1956.
- 2 D. Godfrey and E.E. Bisson, "Bonding of MoS_2 to Various Materials to Form a Solid Lubricating Film," NACA Technical Note 2628.
- 3 F.P. Bowden, Research, vol. 3, 1950.
- 4 D.O. Mitchell, Conference on Lubrication and Wear - 1957, "Wear of P.T.F.E. Impregnated Metal Bearing Materials," London.
- 5 V.G. Fitzsimmons, C.M. Henderson, "Resin-Bonded Teflon Coatings as Dry Film Lubricants," NRL Memorandum Report 777, February 1958.
- 6 B.R. Atkins and D.P. Griffiths, Conference on Lubrication and Wear - 1957, "Electrical Sliding Contacts and Their Behavior at High Altitudes," London.
- 7 E.E. Bisson, R.L. Johnson, and W.J. Anderson, Conference on Lubrication and Wear - 1957, "Friction and Lubrication with Solid Lubricants at Temperatures to 1000°F with Particular Reference to Graphite, London.
- 8 D. Godfrey and E.C. Nelson, "Oxidation Characteristics of MoS_2 and Effect of Such Oxidation on its Role as a Solid Film Lubricant," NACA Technical Note 1882.
- 9 M.B. Peterson and R.L. Johnson, "Factors Influencing Friction and Wear with Solid Lubricants," Lubrication Engineer, vol. 11, no. 5, September through October, 1955.
- 10 M.T. Lavik, "High Temperature Solid Dry Film Lubricants," WADC Technical Report 57-455 Parts I, II, III and IV.
- 11 H.C. Sliney, D.W. Wisander, and R.L. Johnson, "NASA Studies of Solid Film Lubricants for Extreme Temperatures," WADC Technical Report 59-244.
- 12 R.E. Grump, "Solid Film Lubricants," Products Engineering, vol. 27, no. 2, February 1956.
- 13 B. Stupp, "Effects of Surface Preparation on Wear Life of Solid Lubricant Films," WADC Technical Report 59-244.
- 14 W.L. Cox and W.L.R. Rice, "Effects of Nuclear Radiation on Solid Film Lubricants," WADC Technical Report 58-499, January 1959.
- 15 E.W. Breed, and F.V. Morriss, "High Temperature Metallic-Organic and Inorganic Base Fluids," WADC Technical Report 59-203, September 1959.
- 16 E.R. Lamson and M.J. Devine, "High Temperature Lubricants," presented at the 1959 National ACS Meeting.
- 17 W.C. Hart and B. Rubin, "Evaluation of Dry Film Coatings," WADC Technical Report 53-466, Parts I and II, September 1954.