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TECHNOLOGY SURVEY

Technology Utilization Division

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SOLID LUBRICANTS

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Utilization
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SOLID LUBRICANTS

by

M. E. CAMPBELL, JOHN B. LOSER,
AND ELDON SNEEGAS

Prepared under contract for NASA by
Midwest Research Institute
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Foreword

The Administrator of the National Aeronautics and Space Administration has established a technology utilization program for "the rapid dissemination of information . . . on technological developments . . . which appear to be useful for general industrial applications." From a variety of sources, including NASA Research Centers and NASA contractors, space-related technology is collected and screened; and that which has potential industrial use is made generally available. Information from the nation's space program is thus made available to American industry, including the latest developments in materials, processes, products, techniques, management systems, and analytical and design procedures.

This publication is part of a series intended to provide such technical information. It reviews major NASA research and NASA-sponsored research in solid film dry lubrication. This book should be useful to many small companies that have not been acquainted with the growth and use of solid lubricants. Useful references and publications are identified to enable the reader to obtain more specific information. This publication was prepared by the Midwest Research Institute from data supplied by commercial aerospace industry and NASA research organizations.

GEORGE J. HOWICK, *Director*
Technology Utilization Division
National Aeronautics and Space Administration

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Glossary

Antifriction bearing—Usually denotes a ball or roller bearing.

Binder—The material used to hold the pigment of a solid lubricant system 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)—Lubricating oil and grease additives added 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 0 (weakest acid) to 14 (strongest acid).

Hard vacuum—A term used to denote a high vacuum.

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—An abbreviation used to denote 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 wetting ability of the film-forming material. Oiliness is sometimes used as approximately equivalent to lubricity.

Pigment—The solid lubricant material (MoS_2 , graphite, etc.) used in a solid lubricant system.

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

PV—The PV value is the product of load (P) on the projected bearing area (PSI) and the surface velocity in 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 which it has been demonstrated that sliding 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.

TFE—Tetrafluoroethylene.

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

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 per cm². The torr is 1/760 atmosphere, or 1340 dynes per 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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CHAPTER 1

Introduction

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 to 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 new materials developed to improve petroleum oils were first marketed. These materials, called additives, were used to increase the load carrying ability, lubricity, corrosion protection, and thermal oxidative stability of oils.

Since the mid-1930's a strong trend has developed toward using higher and higher temperatures in moving parts. Since the petroleum oils could not adequately do the job at high temperatures, new 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. One result of this trend has been the development and use of solid lubricants to attain the necessary lubrication of delicate parts at extreme temperatures and pressures.

Although the slippery feel and appearance of graphite and molybdenum disulfide have been known for centuries, their use as solid lubricants in the modern sense dates back only about 20 years. Graphite is known by many names, some of which are black lead, carbo mineralis, carburet of iron, caryon noir, plumbago, potelot, reissblei, and silver lead. It was long confused with similar appearing minerals, particularly molybdenite (MoS_2). Not until 1565 was it classified as a separate mineral, and 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 (MoS_2) 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 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. Tables 1, 2, and 3 list other materials that have been considered for use as solid lubricants, as well as coefficient of friction data for those materials.

HISTORY OF SOLID LUBRICATION

Although the use of graphite as a lubricant probably dates back to the middle ages, the use of bonded solid lubricant materials of the type being discussed in this document is relatively new. The first United States patent covering these materials 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 binder being the media by which the lubricating solid is attached to the substrate. Today, approximately 200 patents cover solid lubricating materials and binders.

The first patent issued in this country covered a phosphoric acid bonded graphite. The use of molybdenum disulfide as a lubricating solid also started in the 1940's. It is currently the most popular material. A large variety of binder materials were evaluated in the early bonded film work, including such materials 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 under careful consideration as solid lubricants by a large number of research groups.

The study of solid lubricants, as we know them, is a relatively new field. No systematic study of these materials was begun until long after

TABLE 1.—*Friction Data for New Compounds*

Material	Load (gm)	Coefficient of friction		
		80° F	500° F	1000° F
Ag ₂ Se	326	0.30-0.35	—	0.8-0.9
LiF	"	0.3-0.4	0.9	0.65-0.75
Si ₃ N ₄	"	0.3-0.9	—	0.9
SrS	"	0.7-0.9	—	0.7-0.9
Tl ₂ S	"	0.25-0.5	—	— (melted)
ZnSe	"	0.4-0.6	—	0.55-0.70
ZrN	"	0.2-0.3	—	0.55-0.75
AgI	128	1.0	1.0	1.0
Ag ₂ S	"	0.36	—	—
Ag ₂ Te	"	0.44	—	—
AlPO ₄	"	1.33	1.31	0.31-0.37
AlPO ₄	"	1.0	1.0	0.80
AlPO ₄	326	0.60	—	0.51
BaSO ₄	—	—	—	—
Bi ₂ O ₃	—	—	—	—
Bi ₂ S ₃	128	0.23-0.60	0.21-0.77	0.18-0.38
Bi ₂ S ₃	326	—	0.24-0.39	0.21-0.27
Bi ₂ S ₃	"	0.56-0.58	0.49-0.62	0.20-0.32
Bi ₂ S ₃ + Bi ₂ O ₃	128	0.38	0.20	0.20
Bi ₂ S ₃ + Bi ₂ O ₃	"	—	0.42	0.31-0.37
CaCO ₃	—	—	—	—
CdSe	128	0.23-0.33	0.58	0.27-0.38
CdS	"	0.58-1.0	0.84	0.55
CdTe	"	0.46	0.40	0.33-0.60
CoS	"	0.50-1.15	0.72	0.58
CoSO ₄	326	—	—	—
Cu ₂ S	128	1.0	1.2	1.2
GeO ₂	"	0.48-0.58	—	—
HgS	"	—	—	—
InSe	"	0.46-0.60	0.41-0.60	0.60 (at 750° F)
MoSe ₂	"	0.20-0.33	0.31-0.40	—
NiS	"	0.29-1.0	—	—
PbMoO ₄	"	—	—	—
PbSe	"	0.40-0.67	0.25	0.25
PbS	"	0.08	0.47	0.21
PbS	"	0.30-0.68	—	0.29
PbS(I)	326	0.27-0.39	0.48	0.20
PbS	"	0.47	0.27-0.47	0.15-0.19
PbS-MoS ₂	"	0.16-0.38	0.13	0.37
PbS-Graphite	"	0.20	0.29	0.21
Sb ₂ O ₅	128	0.21-0.96	—	—
Sb ₂ S ₃	"	0.38	0.21-0.49	0.49
Sb ₂ S ₅	"	0.31-0.80	0.35-1.0	—
Sb ₂ S ₅	"	0.50	1.0 (at 300° F)	—
TiTe ₂	128	0.36-0.58	0.95	0.77 (at 670° F)
TiTe ₂	"	0.48-0.58	0.86	0.38-0.48 (at 800° F)
ZnTe	"	0.60-0.68	0.32-0.40	0.58
ZrCl	"	0.45	0.48-0.96	0.27

TABLE 1.—(Concluded)

Material	Load (gm)	Coefficient of friction		
		80° F	500° F	1000° F
Graphite No. 2	128	0.15	0.18-0.22	0.50-0.60
Graphite No. 1	"	0.15	0.10	0.13-0.23
Graphite	326	0.14-0.30	0.06-0.12	0.20-0.27
MoS ₂ + Graphite (7% by weight)	128	0.20-0.25	0.11	0.22-0.56
PbS(II)	—	0.55-0.70	0.3-0.7	—
MoS ₂ + Graphite (31% C by weight)	128	0.16-0.21	0.12-0.14	0.13-0.16
MoS ₂ + Graphite (73% C by weight)	"	0.15-0.21	0.08-0.11	0.11-0.17
MoS ₂	326	0.34	0.10	—

Test Conditions: Mark III Pellet Machine
 Load: 326 g (av. approx. 50 psi)
 Speed: 600 ft/min
 Track: polished, Rex AAA

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. There are a great many areas in which designers should not consider the use of solid lubricants.

TABLE 2.—Results of Lubricity Tests on Thermally Stable Materials

Material	Approximate friction coefficient	
	Room temp.	450° F
BN	0.3	0.15
CdCl ₂	0.6	0.17
CrCl ₃	0.2-0.3	—
PbF ₂	0.6	0.6
MnCl ₂	0.35	0.17
NiCl ₂	0.45	0.19
SnS ₂	0.9-0.45	—
SnO	0.95+	—
SpS	0.95+	0.63
Ta ₂ S ₄	1.15	—
TiC	0.55	—
TiS ₂	0.7	0.6
WS ₂	0.7-1.6	0.2
Almasol	0.7-1.4	—

Test Conditions: Mark III Pellet Machine
 Load: 326 g (av. approx. 50 psi)
 Speed: 600 ft/min
 Track: polished, Rex AAA

TABLE 3.—*Friction of Four Mica-type Minerals*

Material	Chemical Formula	Sample No.	Friction coefficient	
			Air	Vacuum (1×10^{-5} torr)
Pyrophyllite	$[\text{Al}_2(\text{OH})_2]\text{Si}_4\text{O}_{10}$	1	0.51–0.58	0.10–0.17
		2	0.48–0.89	0.10–0.27
Muscovite	$[\text{Al}_2(\text{OH})_2]\text{K Al Si}_3\text{O}_{10}$	1	>1.0	—
		2	>1.0	>1.0
Talc	$[\text{Mg}_3(\text{OH})_2]\text{Si}_4\text{O}_{10}$	1	0.13–0.26	0.21–0.37
		2	0.38–0.72	0.48–0.72
		3	0.55–0.89	0.37–0.65
		4	0.31–0.45	—
Phlogopite	$[\text{Mg}_3(\text{OH})_2]\text{K Al Si}_3\text{O}_{10}$	1	0.38–0.55	—
		2	0.44–0.64	—

NOTE: Friction coefficients determined on Mark VI pellet apparatus:
Load—90 g; Speed—41 cm/sec; temperature—80° F (no heat).

ADVANTAGES AND DISADVANTAGES

Sliney lists the following advantages and disadvantages of solid lubricants (ref. 2).

Advantages

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

Disadvantages

1. Friction coefficient generally higher than with hydrodynamic lubrication.
2. Some wear unavoidable because of solid sliding contact.
3. Dry film coatings have finite wear lives.
4. Solid lubricants have no cooling capacity.
5. Feed rate of gas-entrained solid lubricant powders must be very carefully controlled to prevent clogging and binding with excess lubricant.

Other reports (refs. 3–10) summarized by Benzing (ref. 11) list the advantages as being stable under extreme environments, high temperature, nuclear radiation, low temperatures, very high vacuum, high load conditions, and reactive environments.

Larry Kellogg of Atomic International has indicated that only solid

film lubricants could be used on the moving mechanisms of the AEC's SNAP 8-10 atomic reactors for space experimentation because of the radiation field and temperature extremes involved.

Solid lubricants are often used on components that are difficult to lubricate with conventional liquids. In many cases, the solid can provide lubrication for the life of the part. Solid lubricants can also be used to advantage in such places as safety equipment where an accumulation of dust or dirt cannot be tolerated. Since certain solid lubricants are nonimpact sensitive in LOX, they have found widespread use in this area of application.

It is a complex lubrication engineering job to select the right solid lubricant for a specific application. A lengthy 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. Considerable effort has been expended on basic studies of the lubricating solids, but there is much disagreement among the experts on the lubrication theories covering these materials. (See Bibliography.)

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 where products are named in a list or table, they will be found in alphabetical order.

CHAPTER 2

Bonded Solid Lubricants

Solid lubrication encompasses many separate and distinct areas. This chapter is limited to a discussion of the bonded solid lubricants which comprise probably the largest single area. Although methods of attaching a lubricating solid to a wearing surface are many and vary considerably, the end result is the same, that is, a low friction medium is deposited to reduce friction and wear between two relatively moving surfaces under essentially dry conditions.

The most common lubricating solids, graphite and molybdenum disulfide, have definite limitations for normal operation in air. Molybdenum disulfide (MoS_2) oxidizes at approximately 750°F to MoO_3 , which acts as an abrasive, greatly reducing its performance. For graphite, the limitation is the loss of the absorbed layer of water with increasing temperature, much as in the case of its loss with decreased pressure. Although the materials are adversely affected by high temperature, this is of little concern in normal bonded solid lubricant films because the binders themselves break down before the lubricating solids break down.

RESIN-BONDED SOLID LUBRICANTS

Resin-bonded solid lubricants are probably the most common and widely used solid lubricant types today. This group includes both air-cured and heat-cured materials. These materials usually consist of a lubricating solid called the "pigment" (not to be confused with the pigment in paints) and a bonding agent.

The pigment may be one material or a mixture of several materials. The function of the pigment is to provide the wear reduction and low friction required for the system being lubricated. The binder serves to hold the lubricating pigment to the metal surface so that the motion of the parts does not result in the complete loss of the pigment. The proper pigment-to-binder ratio is very important in the resin-bonded solid lubricants, and varies widely with each manufacturer and with the materials he uses.

Other factors are also important in the overall performance in any given situation. One is the condition of the metal surfaces being lubricated. In most cases the surface is changed or modified by some pretreatment to obtain optimum film performance. Other factors involve the variables directly related to the application of the film, such as spraying techniques which are usually difficult to control.

In addition to application factors, environmental conditions and the operating characteristics of the system being lubricated can drastically affect the film. These factors can modify the desired characteristics of a film and are important in any final selection of material.

The resin-bonded solid lubricants are generally applied in thin films to the surfaces of the metal components being lubricated. In most cases the surfaces have had some form of pretreatment which will depend on the metal substrate being used and the service for which the parts are intended. The resin-bonded solid lubricant films can be applied by spray, dip, or brush. Spraying appears most satisfactory. The coatings applied in this manner are usually 0.0002 to 0.0008 in. in thickness, the optimum being about 0.0005 in. Too thick a film will cause the film to peel or flake off with sliding, whereas too thin a film may result in premature failure due to rupture of the film.

The wear behavior and wear life characteristics of a resin-bonded solid lubricant are different from those of most other lubricants. In its initial wear-in, it will exhibit relatively high wear which will become less with time. The initial high wear 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 is 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 matters investigated. Crump (ref. 1) reports the beneficial effects of hardness on wear rate. Lavik (ref. 2) reports that he 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. Both of these programs were conducted on laboratory simulative bench testers. It has been found, however, that in actual applications the harder bearings offer beneficial results (longer life, lower wear rates), illustrating the dangers of relying only on bench test data.

Bonded dry film lubricants have provided 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

This type of lubricant consists of a lubricating powder, or powders, called the pigment in an air-curing binder material. The lubricating pigment is usually molybdenum disulfide, graphite, or a lubricating plastic such as polytetrafluoroethylene. The resin or binder serves only as a means of improving the adhesion of the powder to the surface to be lubricated. This type of solid film lubricant usually contains a lower total solid content than those listed below as heat-cured. This reduction in solids results in a film with a shortened wear life.

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

Examples of air-drying materials are:

Almasol SFD 10	(Almasol Corporation, Ft. Worth, Tex.)
Lubric-Bond A	{ (Electrofilm Corporation, North Hollywood, Calif.)
Lubric-Bond 35C	
Lubric-Bond J	
Lubric-Bond K	
Molykote M88	{ (Alpha Molykote Corporation, Stamford, Conn.)
Molykote M8800	
Molykote 523Y	
Surf-Kote A-1625	(Hohman Plating and Manufacturing Company, Dayton, Ohio)

HEAT-CURED RESIN-BONDED SOLID LUBRICANTS

In the past, this type of solid lubricant has been the standard of the dry-film lubricant industry. The materials usually consist of the lubricating pigment and a specially formulated resin binder. The lubricating pigment is usually a mixture of approximately 90 percent molybdenum 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 MoS_2 . Films are available that contain small percentages of silver, indium lead, and so forth, as well as a mixture of the MoS_2 and graphite, but the coatings containing only graphite and molybdenum disulfide are more readily available.

The frictional behavior of a material is an important consideration in its selection as a solid lubricant for resin-bonded films. Feng (ref. 3) indicates higher values of friction for a bonded film than for the lubri-

cating 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 300–400° F. Because of the baking temperature, care in the selection of the metal substrate is required. Temperatures above 275° F for 1 hr can be very deleterious to certain aluminum alloys.

Binders that are normally used in the heat-cured solid lubricants are thermosetting and include alkyds, phenolics, epoxies and silicones. Alkyds are relatively inexpensive, cure at low room temperatures, 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 300–400° 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. Normally, they are used only for high temperature service and then only when the brittleness of the silicate type of binder presents a problem.

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° to +450° F.

Examples of the heat-cured materials are:

Almasol SFD 210	(Almasol Corporation, Ft. Worth, Tex.)
Dag. 253	(Acheson Colloids Company, Port Huron, Mich.)
Drilube No. 1	(Drilube Company, Glendale, Calif.)
Everlube 620	(Everlube Corporation, North Hollywood, Calif.)
Lube-Lok 5396	(Electrofilm Corporation, North Hollywood, Calif.)
Molykote X 106	(Alpha Molykote Corporation, Stamford, Conn.)
NPI 14	(National Products Industries, Southgate, Calif.)
Poxylube 500 M	(Poxylube Corporation, Indianapolis, Ind.)
RIA No. 9A	(Sandstrom Products Company, Port Byron, Ill.)
Surf-Kote H 108	(Hohman Plating and Manufacturing Company, Dayton, Ohio)

There are also several custom variations available in these heat-cured solid film lubricants.

PRETREATMENTS

The pretreatment of a metal surface prior to the application of a resin-bonded solid lubricant can greatly affect its performance. Most pretreatments, whether phosphate or the various blasting techniques, tend to increase the surface area and thus give the lubricant binder more area on which to hold. These pretreatments greatly increase the wear life of the lubricant film. Methods used in the pretreating of metals include both chemical and mechanical processes. The type of pretreatment will depend on the base metal being employed. (Such pretreatments are required in nearly all of the solid lubricant specifications described in Chapter 3.)

A list of pretreatments for various metal surfaces to improve wear-life of resin-bonded solid lubricants follows:

<u>Type of Metal</u>	<u>Pretreatment</u>
Steel—except stainless	Grit blast or other mechanical process Phosphating Sulfiding
Aluminum	Anodize Chemical film
Chromium and nickel plating	Vapor grit or sand blast
Titanium	Vapor or grit blast
Cadmium plate	Phosphating
Zinc plate	Phosphating
Copper and copper alloys	Bright dip
Magnesium	Dichromate treatment
Stainless steel	Vapor or grit blast Chemical etch

There are probably many other pretreatments that might 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 many workers in the field. Devine et al. (ref. 4) 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 large reservoirs machined in a metal surface also give improved performance. Investigators at the Boeing Company 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 mechanical blasting. These have had widespread use, particularly in the aerospace industries where common bearing or wearing surfaces are steel, aluminum, stainless steels, titanium, and others. In the past, the phosphate coatings have been used extensively with liquid lubricants to improve bearing life and to gain a small measure of corrosion protection. They are used under solid lubricants for the same purpose to gain some corrosion protection, since the solid lubricants do not provide such protection. Likewise, anodizing is preferred on aluminum parts to obtain the necessary corrosion protection.

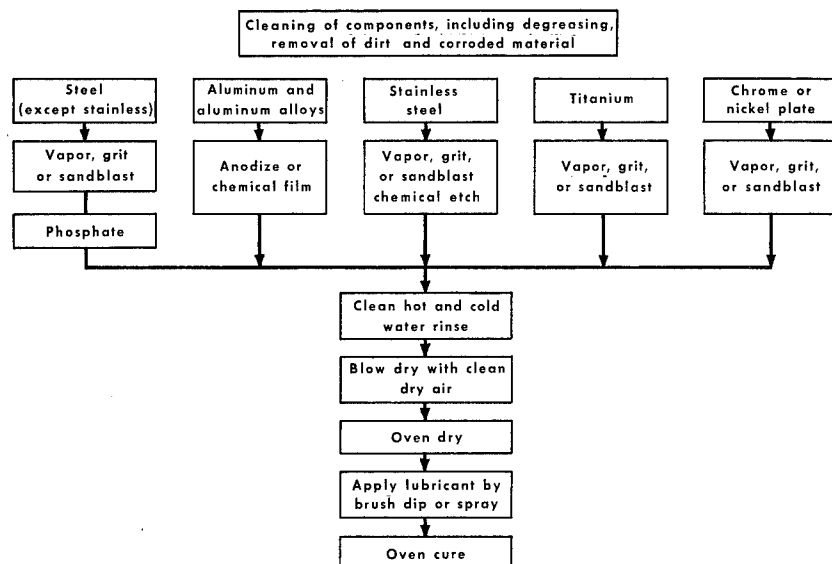
The proper application of phosphate pretreatments is a difficult procedure. Stupp (ref. 5) reports on a detailed investigation involved in obtaining a good phosphate coating. The effects of bath temperature, immersion time, and bath acid strength on the thickness of the coating, as well as the structure and size of crystal growth, are noted. Stupp found that the best conditions were: bath temperature of 205° F, bath acid 7 to 8 points (full acid), and a treatment time of approximately 15 min. M. E. Campbell of North American Aviation is in complete agreement with this investigator because he has too often seen faulty solid lubricant coatings resulting from an improperly controlled phosphate bath. An important consideration in the use of phosphate treatment is the temperature to which the part will be subjected in use. Phosphate coatings are not recommended for use at over 600° F, as has been demonstrated by Lavik (ref. 6).

APPLICATION PROCESS

The processes by which the solid lubricants are applied to metal surfaces can also have considerable effect on the film behavior and performance. The actual application of the lubricant by spraying, dipping, and brushing has already been briefly mentioned. These items are only one step in a large overall process. Of the three common methods, spraying and dipping are most often used. However, there certainly are cases where brushing can be used to advantage. The commonly held opinion that "spraying a dry lubricant is just like spraying paint," is not correct. A dry lubricant is a very special material and should be treated as such. Applying a solid lubricant in the sloppy manner often used in spray painting will result in a very degraded film. In a dipping process, the entire part is usually completely immersed in a lubricant bath.

Table 4 shows general processes for applying resin-bonded solid lubricants to various metal surfaces. Similar processes are outlined in specifications, detailed in Chapter 3, sections C., D., and H. Most manu-

facturers of bonded solid lubricants have processes that they recommend for their particular films. Although most process specifications are similar, they vary as to condition of time and temperature for curing of the films, because of the variations in materials used in the preparation of the films.

TABLE 4.—*Application Process*

Notes: 1. Parts to be dry-lubricated should be handled only with clean, white cotton gloves after pretreatment operation.

2. Continuous inspection required during various processing steps.

3. Dry-lubricated parts should be packaged separately as soon as possible after curing.

We have attempted to show that close control of the many variables is important. Accurate control of such items as phosphating baths is essential, and the proper consistency of material to be sprayed must be maintained. The latter can be maintained by the addition of proper thinners to the lubricant mixture prior to application on parts. Other variables also are important, including the curing temperatures. 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 5 gives suggested maximum curing temperatures and times for several materials. An under-cured film will have poor adhesion and fluid resistance, while the coating cured for too long at an extremely high temperature will probably have a reduced wear life because the binder will char, thereby destroying the adhesion to the substrate material.

The actual equipment used in the application process can be simple

TABLE 5.—*Suggested Baking Schedules for Resin-bonded Solid Lubricants*

Substrate Material	Baking Schedule	
	Temperature (° F)	Time (min)
Steel and alloys	400	60
Steel and alloys case hardened	300	60
Stainless (except 440)	400	60
440 stainless steel	300	60
Aluminum and magnesium alloys	275	60
Aluminum and magnesium alloys	300	25
Bronzes	300	60
Nickel and nickel alloys	400	60
Titanium	400	60
Monel and Inconel	400	60
Plated surfaces (chrome or nickel plated)	400	60

or very complex. The type of application process used will depend on the quantity and size of parts to be coated.

In the opinion of nearly all large users of bonded solid lubricants, careful pretreatment and processing of a bonded solid lubricant will result in a good quality film.

INORGANIC BONDED SOLID FILM LUBRICANTS

Materials of this type 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 1200° F. There is some considerable overlap in their applicable temperature ranges, and certain ones operate very satisfactorily at temperatures down to -300° F. The high temperature inorganic solid lubricants are a logical extension to the resin-bonded films. 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. Dry lubricants of this type usually contain lubricating solids (pigments) such as lead oxide, lead sulfide, calcium fluoride, gold, silver, tellurides, selenides, and so forth. There are exceptions, however, and a number of the ceramic and salt-based binders are used with MoS₂ and graphite.

Because the inorganic-bonded solid lubricants encompass two totally different bonding media, nonceramic and ceramic, they will be discussed separately.

NONCERAMIC (SILICATE, PHOSPHATE)

The bonding technique for these materials commonly employs water-soluble silicates, phosphates, etc., which produce a hard coating that tends to be somewhat brittle when cured. In general, they can be used

at temperatures from -300° to $+1000^{\circ}$ F. Solid lubricants containing the salt-based binders usually contain graphite, MoS_2 , lead sulfide, powdered metals, etc. They can be used in extremely high load areas, with loads in excess of 100,000 psi. However, in applications where movement is a prime design consideration, their wear life is not as good as resin-bonded films. They have two advantages: they are not subject to outgassing in a vacuum of 10^{-9} torr, and they are compatible with liquid oxygen. Both these advantages have made them useful lubricants in the space programs. But, along with these advantages, they have certain drawbacks: they do not afford corrosion protection, and the film will soften when subjected to water or moisture for extended periods of time. (Because certain of these films are considered new developments, they will be discussed at greater length in Chapter 4.)

Examples of salt-based lubricants are:

Almasol SFD 570	(Almasol Corporation, Ft. Worth, Tex.)
AML-23A	(Naval Air Material Aeronautical Materials Laboratory, Philadelphia, Pa.)
Drilube 805	(Drilube Company, 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.)
X-15	(Alpha Molykote Corporation, Stamford, Conn.)
811	(Everlube Corporation, North Hollywood, Calif.)
2306	(Electrofilm Corporation, North Hollywood, Calif.)

The pretreatments for resin-bonded solid lubricants listed previously are also employed in conjunction with the inorganic non-ceramic bonded lubricants. However, the most accepted pretreatments for these films are the vapor or grit blast methods. Since these films are used at elevated temperatures, the chemical pretreatments are not recommended because of their temperature limitations.

Application of the inorganic non-ceramic films is similar to that employed for the resin-bonded materials shown in table 4. Since certain

of these films contain heavy metal powders, constant agitation is required during application. They can be sprayed, dipped, or brushed as desired. Baking temperature is usually in the same temperature range as the resin-bonded materials, but is often a two-step process, an example being 180° F for 2 hr followed by a bake at 400° F from 2 to 16 hr.

CERAMIC-BONDED

The ceramic bonding agents are glasses rather than resins, and melt on heating. On cooling they serve as a bonding agent for the dispersed lubricant. Their principal advantage is their good strength at elevated temperatures.

The lubricating solids commonly employed with ceramic binders are graphite, CaF_2 , lead oxide, and so forth. 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 as the resin-bonded materials at lower (room) temperatures, they generally exceed the resin film's capability by a considerable amount (over 10 times) at higher temperatures. There are exceptions, however, one being when the lubricants are run at high speeds which result in high temperatures being generated in the contact zone. In such case, the ceramic-bonded films will generally out-perform the resinous films. North American Aviation has shown that their Vitrolube material does out-perform the resin-bonded lubricants at room temperature (at low speed as well as high speed).

Films of the ceramic-binder type that are available include:

Electrofilm 1000	(Electrofilm Corporation, North Hollywood, Calif.)
Vitrolube	(North American Aviation Company, Inc., Los Angeles, Calif.)
NASA CaF_2	(National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio)

In the application of all types of bonded solid lubricants, especially the resin and silicate types, pretreatment of the base substrate is very important, particularly if maximum adhesion, long wear life, and low friction are required.

In general, the substrate material or substrate pretreatment has a great effect on the wear life of any film. These effects are probably due to many different factors, two of which are the hardness of the base metal and the degree of bonding, which are important with the resin-bonded materials. With the ceramic materials, pretreatment in nearly

all cases is of the sand blast variety. However, Sliney (ref. 7) reports that a period of oxidation at 2000° F on René 41 alloy improves the life of the NASA calcium fluoride film.

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 expansion characteristics are not the same or very similar, the coating will be fractured and be easily removed from the substrate.

Application of the ceramic-bonded lubricant films is by the usual methods—spraying, dipping, or brushing. Since these are heavy materials in a water slurry, care must be exercised to insure that they are properly mixed at all times during application.

CHAPTER 3

Specifications

The widespread use of dry, solid lubricant powders, thin film-bonded lubricant coatings, and reinforced plastic materials discussed in Chapter 2 has resulted in a number of military and industrial specifications being written covering their application and use. The most notable are the current military specifications being used by all government agencies. This list includes, but is not necessarily limited to, the following specifications:

A. MIL-M-7866 MOLYBDENUM DISULFIDE, POWDER, LUBRICANT

This specification covers one grade of powdered molybdenum disulfide, to be used for the lubrication of sliding surfaces where boundary conditions exist.

USES

Intended for use as a dry lubricant or as a component with a suitable specification oil or grease for special applications, where no other specification lubricant is satisfactory. It is used for thread anti-seize, for lightly loaded applications where a fluid lubricant is objectionable, and occasionally with approved oil or grease for highly loaded gears where the specification oil or grease is not adequate.

LIMITATIONS

The bonded lubricant does not give corrosion protection. This material must be bonded to surface to utilize maximum lubrication potential. Mixtures of this powder with oils or greases should not be prepared in the field or for applications where performance data have not been established.

B. MIL-G-6711 GRAPHITE, LUBRICATION

USES

This material is a 200-mesh, high grade, powdered graphite for use either as a dry lubricant or mixed with a suitable proven specification

oil. MIL-M-7866 molybdenum disulfide should be used in preference to this material, if possible.

LIMITATIONS

This material is not intended for blending with oils or greases.

C. MIL-L-8937 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.

USES

This solid film lubricant is intended for sliding motion types of applications such as plain spherical bearings, flap tracks, hinges, cam surfaces, gears, and so forth.

LIMITATIONS

This material should not be used with oils or greases unless experience indicates otherwise. Since this material requires a bake at 300° F for 1 hr, it should not be applied to materials whose properties are adversely affected by exposure to these conditions.

CONDENSED SPECIFICATION REQUIREMENTS

Film Appearance and Thickness.—The bonded solid film lubricants, when examined in accordance with the applicable sections of (ref. 1), shall appear uniform in color, smooth, and free from cracks, scratches, pinholes, blisters, bubbles, runs, sags, foreign matter, grit, rough particles, separation of ingredients, or other imperfections.

Film Adhesion.—The bonded solid film lubricant, when tested in accordance with applicable sections of (ref. 1), shall not be lifted from the test panel by the tape employed. A uniform deposit of powdery material may cling to the tape, but lifting of any flakes or particles of the lubricant which exposes any bare metal shall indicate unsatisfactory adhesion.

Thermal Stability.—The bonded solid film lubricant shall not flake, crack, or soften, and shall exhibit satisfactory adhesion when tested at —65° and 500° F in accordance with the applicable sections of (ref. 1).

Fluid Resistance.—The bonded solid film lubricant shall not soften, lift, blister, crack or peel, and shall exhibit satisfactory adhesion when half immersed for 24 hr at room temperature in each fluid tested, when evaluated in accordance with the applicable sections of (ref. 1).

Endurance Life.—The bonded solid film lubricant, when tested at the Falex lubricant tester in accordance with applicable sections of (ref. 1), shall have an average endurance life of at least 60 min at 1000 lb gage load. The endurance life of any single run shall not be less than 50 min. Four tests are required.

Load-Carrying Capacity.—The bonded solid lubricant must demonstrate a load-carrying capacity of at least 2500 lb gage load on the Falex lubricant tester when examined in accordance with applicable sections of (ref. 1). Duplicate tests are required.

Corrosion Resistance.—The bonded solid film 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 applicable sections of (ref. 1).

D. MIL-L-23398 LUBRICANT, SOLID FILM, AIR DRYING

This specification establishes the requirements for an air-drying solid film lubricant intended to reduce wear and prevent seizing and galling.

USES

This dry solid film lubricant is intended for bonding on steel, titanium, aluminum and aluminum alloys where conventional lubricants are difficult to apply. This is an air-dried material which can be applied to parts of complete assemblies where curing by heating is not possible, such as plain bearings, flap tracks, hinge pins and cam surfaces. The material will provide a low friction surface to reduce wear and prevent galling and seizing of rubbing parts.

LIMITATIONS

This material should not be used with oils or greases unless experience indicates otherwise. Spraying should be done with adequate ventilation. Caution must be exercised to prevent overheating of spray cans, or use in areas where potential ignition sources exist. This material should not be substituted for MIL-L-8937, since it is inferior in wear life and load carrying ability.

CONDENSED SPECIFICATION REQUIREMENTS

Film Condition.—The bonded solid film lubricant shall be uniform in color, smooth, and free from cracks, scratches, blisters, foreign matter, grit, rough particles, bubbles, pinholes, runs, sags, or other surface imperfections, and shall show no evidence of separation of material ingredients when examined in accordance with applicable sections of (ref. 2).

Film Adhesion.—The bonded solid film lubricant shall not be lifted from the test panel by the tape test described in the applicable section of (ref. 2). A uniform deposit of powdery material may cling to the tape, but the lifting of any flakes or particles of the lubricant that exposes bare metal shall indicate unsatisfactory adhesion.

Thermal Stability.—The bonded solid film lubricant shall not flake,

crack, or soften, and shall show satisfactory adhesion after exposure to 500° F for 3 hr, followed by 3 hr at -65° F in accordance with applicable sections of (ref. 2).

Endurance Life.—The bonded solid film lubricant shall have a minimum average wear life of 60 min when tested on the Falex lubricant tester at 1000 lb gage load in accordance with applicable sections of (ref. 2). Four tests are required, and the wear life of any single run shall not be less than 50 min.

Load-Carrying Capacity.—The bonded solid film lubricant shall have a load-carrying capacity of at least 2500 lb gage when tested on the Falex lubricant tester in accordance with applicable sections of (ref. 2). Duplicate runs are required.

Fluid Resistance.—The bonded solid lubricant shall not flake, crack, or peel, and shall exhibit satisfactory adhesion after being immersed for 24 hr at room temperature in the fluids specified in the specification when tested in accordance with applicable sections of (ref. 2).

Corrosion Resistance.—When subjected for 500 hr to the heat and humidity conditions described in (ref. 2), the bonded solid film lubricant shall not show or cause discoloration, pitting, formation of white corrosion deposits, or other evidence of corrosion deposits or corrosion.

E. MIL-G-21164 GREASE, MOLYBDENUM DISULFIDE (FOR LOW AND HIGH TEMPERATURES)

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 should not be used for other than steel surfaces without prior performance evaluation.

F. MIL-L-25681 LUBRICATING OIL, MOLYBDENUM DISULFIDE, SILICONE BASE, HIGH TEMPERATURE

USES

This material is intended for use on slow-speed sliding surfaces operating at temperatures up to 750° F and for use as an anti-seize compound on threaded parts which operate at temperatures up to 1400° F.

LIMITATIONS

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

G. MIL-L-22273 AND MIL-L-25504

Both specifications cover a heat-cured dry lubricant. They have since been replaced by MIL-L-8937, item (C) above.

H. MIL-L-46010 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. The lubricant covered by this specification does not contain graphite or powdered metals.

USES

Same as MIL-L-8937.

LIMITATIONS

Same as MIL-L-8937.

The corrosion protection of this film is probably obtained only by the phosphate coating applied to the base material.

CONDENSED SPECIFICATION REQUIREMENTS

Film Thickness.—The lubricant shall be capable of being sprayed, brushed, or dipped and cured to a film thickness of 0.0002–0.0005 in. All thickness measurements must fall within the above limits when determined in accordance with applicable sections of (ref. 3) and ref. 4b).

Wear Life.—The cured lubricant shall provide an average minimum Falex wear life of 450 min at a 1000 lb gage load when tested in accordance with applicable sections of (ref. 3) and (ref. 4a). Four tests are required. No single test shall provide a wear life of less than 390 min.

Load-Carrying Capacity.—The cured lubricant film shall provide an average minimum Falex load-carrying capacity of 2000 lb gage when tested in accordance with applicable sections of (ref. 3) and (ref. 4c). Duplicate tests are required. No single test shall provide a load-carrying capacity of less than 1750 lb gage.

Corrosion Protection.—The cured lubricant film, when applied to test panels to a thickness of 0.0002–0.0005 in., shall show a maximum of three round dots per panel after a minimum salt spray exposure of 100 hr in accordance with applicable sections of (ref. 3) and (ref. 4d).

Film Adhesion.—The cured lubricant film shall not be lifted from the test panel by the tape employed. A uniform deposit of powdery material may cling to the tape, but the lifting of flakes or particles of lubricant which exposes only bare metal shall indicate unsatisfactory adhesion. Adhesion tests are to be conducted in accordance with applicable sections of (ref. 3) and (ref. 4b).

Fluid Resistance.—The cured lubricant film must demonstrate adequate adhesion as described above after immersion in selected fluids at 74° F for 24 hr.

High and Low Temperature Stability.—The cured lubricant coating must demonstrate adequate adhesion after having been soaked at 500° F for 4 hr. It must also demonstrate adequate adhesion after soaking on a cake of carbon dioxide ice for a period of 4 hr.

Graphite and Powdered Metals.—The lubricant shall contain no graphite or powdered metals when tested in accordance with the applicable section of (ref. 3).

I. MIL-L-81329, LUBRICANT, SOLID FILM, EXTREME ENVIRONMENTS

This specification establishes the requirement for a solid film lubricant to be used to extreme environments, including temperatures ranging from -300° to +1000° F liquid oxygen and vacuum to reduce wear and prevent galling and seizing of metal surfaces.

CONDENSED SPECIFICATION REQUIREMENTS

Materials.—Materials shall consist essentially of lubricating solids in a suitable binder, at spraying consistency. Organic materials are not suitable for lubricant compositions furnished under this specification. The applied film shall be capable of being cured within ½ hr at 77° ± 3° F, 2 hr at 180° F, and 2 hr at 300° F.

Flammability.—The lubricant shall be nonflammable when tested, as described in (ref. 5).

Appearance and Thickness.—When examined in accordance with (ref. 5), the solid film lubricant shall be free of surface imperfections and shall show no evidence of separation of material ingredients. The thickness of the finished film shall be 0.0002-0.0005 in.

Film Adhesion.—The bonded film lubricant, when tested in accordance with applicable sections of (ref. 5), shall not be lifted from the test panel by the tape employed. A uniform deposit of powder may cling to the tape, but the lifting of particles of lubricant which exposes any bare metal shall indicate unsatisfactory adhesion.

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

Endurance Life.—The solid film lubricant shall demonstrate an average endurance life of at least 100 min when tested on the Falex lubricant tester at 1000 lb jaw load. Four tests shall be conducted. The endurance life of any single run shall not be less than 80 min.

High Temperature Performance.—The solid film lubricant when tested in accordance with (ref. 5) and (ref. 6) shall demonstrate a

useful life of 500 hr at 750° F and 10 000 rpm continuous running on M-10 steel, SAE 204 size bearing with an ABEC-3 tolerance.

Vacuum Performance.—The solid film lubricant, when tested in accordance with applicable sections of (ref. 5), shall demonstrate a performance life of 100 hr at 1000° F in a vacuum of 1×10^{-6} torr.

Shock Sensitivity with Liquid Oxygen.—The solid film lubricant shall give no reactions in 25 test drops at 70 ft-lb of impact.

J. MIL-B-8942 BEARINGS, PLAIN, TFE LINED, SELF-ALIGNING

Although this is not a dry lubricant specification, it covers a bearing lubricant package, and the lubricant is essentially a dry lubricant.

The specification covers plain spherical bearings which are self-aligning and self-lubricating by incorporating tetrafluoroethylene (TFE) in a liner between the inner ring (ball) and outer ring (race) for use in the temperature range -65° to $+250^{\circ}$ F.

K. MSFC-253 LUBRICANT, DRY FILM, CERAMIC MLF-5, SPECIFICATION FOR

This specification applies to a dry film lubricating material, designated MLF-5, which has a low coefficient of friction, is capable of supporting high loads without penetration, and which is applied to a substrate by air-brush spraying to form a thin coating.

L. MARSHALL SPACE FLIGHT CENTER SPECIFICATION NO. 81205, "BONDED SOLID DRY FILM LUBRICANTS," MATERIAL SPECIFICATION FOR, AND APPLICATION OF "BONDED SOLID DRY FILM LUBRICANTS"

The above two NASA specifications cover the quality of the solid lubricant material as well as the application procedures.

There are several types of dry lubricants specified on the above specifications, including a Type I for general purpose use from -100° to -475° F; Type II, "Cryogenic, Wide-Temperature Range" for operation at -150° to 1100° F; and Type III, "Cryogenic, Wide-Temperature Range and LOX-Compatible" for operation from -320° to $+650^{\circ}$ F.

A word of caution is offered in regard to the use of the afore-discussed specifications. They are not cure-alls, nor are the materials qualified to them. However, when used wisely, they can be very helpful in the selection of solid film lubricants for specific applications.

CHAPTER 4

New Developments

The development of bonded solid lubricants has not always kept pace with the ever changing requirements of industry. This is 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, compatibility with oxidizers, high load carrying ability, long wear life, and many others.

Many Federal Government agencies, including the Army, Navy, Air Force, NASA, and the FAA, are sponsoring research work both in-house and on-contract, that is directed toward the development of new solid lubricants and techniques of lubrication. Many large aircraft companies also have such programs. This research work has yielded many significant contributions, and some new solid lubricants are now developed to the point where they are usable materials. We cannot include all such materials in a publication of this nature, but the remainder of this chapter will be devoted to a discussion of five of these materials.

LUBECO 905

Very little technical information is available about this material, but it is known to contain an inorganic type binder. The manufacturer considers it to be proprietary.

The material has been evaluated by the NASA Marshall Space Flight Center, where it has successfully passed their high load test of 150 000 psi, and is reported to be in use extensively on the Apollo space capsule.

MLF SERIES

Midwest Research Institute in Kansas City, Mo., has been conducting a program for NASA's Marshall Space Flight Center on Contract NAS8-1540 to develop, investigate, and evaluate solid lubricant films suitable for use in both normal atmosphere and space environ-

ments. Over the past several years, the bonded solid lubricant films listed in table 6 have been developed on this program.

TABLE 6.—*Solid Lubricant Film Compositions and Specifications*

Film designation	Lubricant-to-binder ratio	Ingredients and percent solids by weight
MLF-5	10:1:5/7	MoS ₂ (51), graphite (5), gold (26), sodium silicate (18)
MLF-6	10:1:3/7	MoS ₂ (57), graphite (6), bismuth (17), sodium silicate (20)
MLF-7	10:1:5/5	MoS ₂ (51), graphite (5), gold (26), potassium silicate (12), sodium fluoride (6)
MLF-8	10:1:5/8	MoS ₂ (50), graphite (5), gold (25), aluminum phosphate (20)
MLF-9	10:1:14/9	MoS ₂ (34), graphite (4), bismuth (47), aluminum phosphate (15)

SPECIFICATIONS

MoS₂—Type Z, Alpha Molykote Corporation, Stamford, Conn.

Graphite—No. 635, Joseph Dixon Crucible Company, 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.

Sodium Silicate—Type K, Philadelphia Quartz Company, Philadelphia, Pa.

Aluminum Phosphate—50 percent solution, Stauffer Chemical Company, New York, N. Y.

The amount of development and testing already conducted on the films listed in table 6 is so voluminous that it is impossible to report it in its entirety here. However, the complete history of these materials can be found in references 1, 2, and 3.

NASA—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. 4).

In the 1956–1958 period, they became involved in the development of lubricants for use in the 400° to 1000° F temperature range. In one such investigation (ref. 5), lead oxide (PbO) was tested but found to be an effective lubricant only in the range of 500° to 1250° F. Because the organic type binder materials were unsatisfactory at these temperatures, silicon dioxide (SiO₂) was used as a binder on the PbO film. At 1250° F, this coating had a coefficient of friction that varied from 0.10–0.25, 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. Testing on this film and on the CaF₂ film described later was accomplished on equipment similar to that described in Chapter 6 and shown in figure 37.

The PbO-SiO₂ film did not become popular in the solid lubricant industry. However, 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. 6). They required that the material 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. They concluded that of the materials investigated only CaF₂ merited further investigation.

Along with the investigation to determine the best lubricity solid, a program to develop binder materials was also conducted. Friction coefficients for the ceramic coatings were found to be between 0.3 and 0.4 over the range of 75° 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 film (ref. 7) has now been patented (ref. 8).

The ceramic-bonded CaF₂ lubricant film is recommended for use on nickel alloys over the temperature range of 500° to 1900° F in both air atmosphere and hard vacuum. The material can be thinned with water and applied by spraying, brushing, or dipping. Recommended coating thickness is 0.0008 to 0.0035 in., with from 0.001 to 0.002 in. 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

TABLE 7.—*The Binder*

(All figures in percent by weight)			
	Suitable range	Preferred range	Most preferred
Cobalt oxide (CoO)	40-70	55-65	60
Barium oxide (BaO)	10-30	15-25	20
Boric oxide (B ₂ O ₃)	10-30	17-23	20

best endurance life to the film. An overlay of pure CaF_2 also increases wear life but tends to increase the coefficient of friction.

According to reference 8, the composition of the films should be as shown in table 7.

The pigment (lubricating solid CaF_2) to binder ratio can vary within wide limits. The most preferred ratio is 3:1. The effects of binder content on friction and wear at various temperatures are given in table 8.

TABLE 8.—*Binder Content Effects on Calcium Fluoride Solid Lubricant*

Effect of ceramic content on coefficient of friction				
	500° F	1000° F	1200° F	1500° F
1 part binder to 9 parts CaF_2	—	0.16	0.08	0.10
1 part binder to 4 parts CaF_2	0.30	0.18	0.07	0.08
1 part binder to 3 parts CaF_2	0.22	0.12	0.11	0.15
1 part binder to 1 part CaF_2	0.30	0.16	0.35	0.20
4 parts binder to 1 part CaF_2	0.49	—	—	0.15
Effect of ceramic content on endurance life (in number of test cycles to failure).				
	500° F	1000° F	1200° F	1500° F
1 part binder to 9 parts CaF_2	—	14 000	38 000	17 000
1 part binder to 4 parts CaF_2	2 000	21 000	19 000	17 000
1 part binder to 3 parts CaF_2	(1)	(1)	(1)	29 000
1 part binder to 1 part CaF_2	24 000	(1)	25 000	28 000
4 parts binder to 1 part CaF_2	3 000	—	3 000	43 000

Note: (1) No failure after 50 000 cycles.

RIA #9A

This material was developed by the U.S. Army at the Rock Island Arsenal in Rock Island, Ill. Several papers and reports have been published concerning the development of this material (see refs. 9, 10, and 11). It is known to contain an oxide of a group five metals (columbium, vanadium, etc.) and a metal phosphate. The primary lubricating material is molybdenum disulfide. The binder material is a mixture of 75 percent phenolic and 25 percent epoxy resins.

Specification MIL-L-46010, described in Chapter 3, has been written covering this material. The advantage of the material appears to be its good corrosion qualities which are reported to be far better than any other resin-bonded lubricant. Whether this is gained from the additives incorporated in the material or the rather stringent pretreatments spelled out in the specification is not known.

Murphy and Mead (ref. 9) have made a comprehensive list of applications where the material is being used by the Army Ordnance Corps. The Rock Island Arsenal does not supply this material commercially,

but it is available from the Sandstrom Products Company, Port Byron, Ill.

VITROLUBE

Vitrolube is the trade name of a ceramic-bonded solid lubricant developed by North American Aviation Company (ref. 12) to withstand the stringent load and temperature conditions encountered during the operation of the XB-70A, Mach 3 aircraft. The Vitrolube material contains molybdenum disulfide and graphite in the ratio of 7:2. The glass (ceramic) binder is a combination of glasses which have 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 complete description of Vitrolube, including tables and charts of its properties, is included in the Appendix, along with an account of the development and testing of this lubricant.

CHAPTER 5

Other Solid Type Lubricants

Thus far, only the thin film bonded solid lubricants have been discussed. Several others that may be of equal importance are finding wide use in many industries. This section will briefly discuss a number of these materials.

LUBRICATING SOLIDS AS ADDITIVES

Lubricating solids are added to oils and greases to improve their lubricity and EP characteristics. The lubricating solid additive tends to prevent or delay seizure in places where the film of oil or grease is disrupted. The military, automotive, and heavy electrical industries are using large quantities of these materials. The Navy uses in excess of 100 000 lb each year in aircraft, catapults, and other shipboard machinery. Certain automotive companies specify such a lubricant for ball-joint applications. The heavy electrical industry uses these materials 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 how much additive is 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 as an additive 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 lubri-

* The authors are indebted to C. R. Bower of E. I. duPont de Nemours and Company for this section of "Lubricating Plastics."

cation requirements of bearings. Properly designed nonlubricated plastic bearings can often solve this problem. These nonlubricated plastic bearings can also help eliminate problems resulting from: high wear rate, lubricant failure, chemical corrosion, stick-slip motion, inaccessibility for lubrication, and high or low operating temperatures.

BEARING TYPES AND METHODS OF FABRICATION

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, as shown in figure 1.

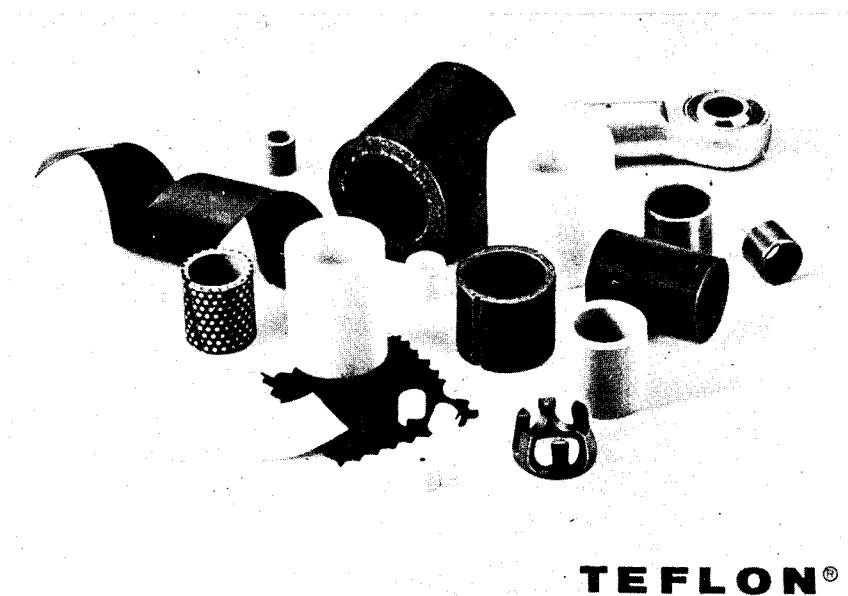


FIGURE 1.—Bearing configuration available in plastic bearings.

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, as shown in figure 2. These bearing materials can be manufactured by injection molding, compression molding, or machining of basic shapes. The final configuration and material selection will determine the most suitable means of fabrication.

MATERIALS OF CONSTRUCTION

Nonlubricated plastic bearings usually are fabricated from nylon, acetals, tetrafluoroethylene, polyimides, and phenolics. These materials

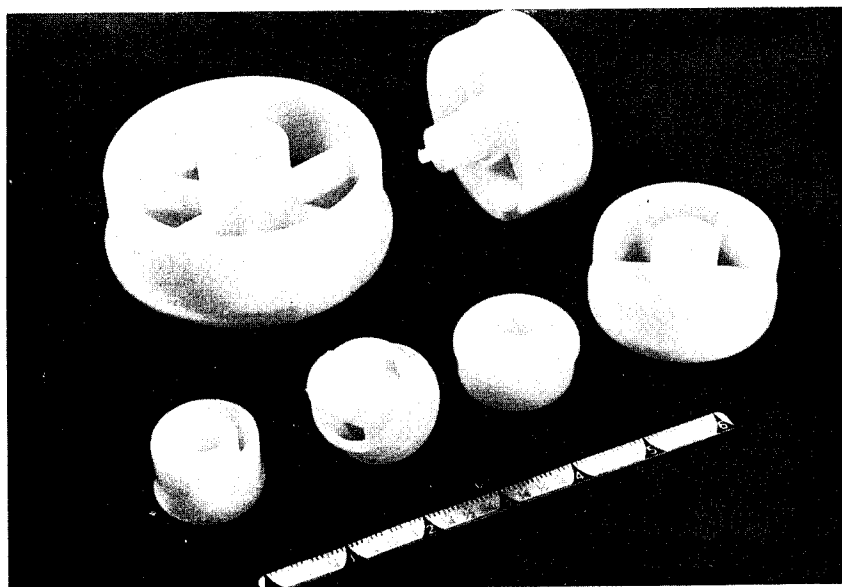


FIGURE 2.—Conveyor roller end caps of duPont zytel nylon molded resin.

are often compounded or blended with other 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 25 μ in. or finer; chromium plating tends to reduce wear further.

Glass fiber fillers in nylon reduce cold flow and thermal expansion, but increase the coefficient of friction. Friction and wear are reduced by dry lubricant fillers such as molybdenum disulfide, graphite, and TFE fiber. 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 frictional coefficient against a steel shaft is comparable to nylon.

Powdered TFE resin or molybdenum disulfide 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 wear resistance.

Because of their relatively low cost and good structural properties, acetals are often used as mechanical parts or housings; if a bearing surface is needed, unfilled acetals offer good bearing performance without added parts.

TFE has the lowest friction of any plastic, and its coefficient of friction decreases with load. This resin has the highest resistance 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.

Fillers increase load-carrying capacity and reduce the size of the bearing, 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 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 fpm. 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 is impregnated with a mixture of TFE resin and powdered lead. 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 0.05–0.1; at speeds of 1000 fpm, the coefficient nearly doubles.

Teflon TFE fibers have extremely high load-carrying ability. In a woven form they can support loads up to 40 000 psi. Voids in the fabric and thin layers eliminate the thermal expansion problem 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 their method of construction. These fibers are usually bonded to a backing, and as the temperature goes up the bond tends to weaken. This limits the surface speed to about 100 ft/min.

Phenolic plastic bushings have good resistance to cold flow, have high strength and high service temperature, but have higher friction and poor resistance to abrasion. However, 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

* The PV value is the product of load on the projected bearing area (psi) and the surface velocity (ft/min).

TABLE 9.—Comparative Performance Data for Self-Lubricating Plastic Bearing Materials

	Modified polymers				Unmodified polymers					
	Nylon	Acetal	Fluoro-carbons	Poly-imides	Phenolics	Nylon graphite filled	Acetal TFE fiber filled	Fluoro-carbon wide range of fillers used	Polyimide graphite filled	Phenolic TFE filled
Properties	Maximum load, projected area, zero speed, psi	5200	1000	>10,000	>4000	>1000	1800	2000	>10,000	>4000
	Speed, max fpm*	500	100	>1000	1000	200-400	800	>1000	>1000	>1000
PV for continuous service, 0.005 in. wear in 1 000 hr, psi = fpm	1000	1000	200	300	100	1000	2500	2500 50,000**	3000	5000
Limiting PV at 100 fpm, psi = fpm	4000	3000	1800	>100,000	5000	4000	5500	30, 000	>100,000	>40,000
Friction Coefficient	0.20-0.40	0.15-0.30	0.04-0.13	1-3	0.90-1.1	0.1-0.25	0.05-0.15	0.04-0.25	1-3	0.05-0.45
Wear Factor, K x 10 ⁻¹⁰ cu in. min/ft-lb-hr	50	50	2500	150	250 2000	50	20	1-20	15	10
Elastic modulus, bending, psi x 10 ⁶	0.3	0.4	.08	0.45	5	0.4	0.4	0.4	0.63	5
Critical temp at bearing surface, °F	400	300	500	>600	300-400	400	300	500	600	300-400
Thermal conductivity btu/in/hr/sq ft/F	1.7	1.6	2.2	2.2				3.0-5.6	2.2	
Thermal expansion in./in./F x 10 ⁶	55	45	80	30	25	35-50	45	50	30	25
Resistance to humidity	fair	good	excellent	good	good	fair	good	excellent	good	good
Resistance to chemicals	good	good	excellent	good	good	good	good	excellent	good	good
Density, gr/cc	1.2	1.43	2.15-2.20	1.42	1.4	1.2	1.54	2.15-2.25	1.49	1.4
Cost index for base material	1.4	1	5	15		1.5	6	5	15	

*Continuous operation under 5 lb load.

**Exceeds Limiting PV.

arranged radially serve as wicks for the oil; when the bearing warms up and the lubricant becomes a fluid, 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. Wall thickness is at least $\frac{1}{4}$ in.

Polyimides have the highest PV limit of any organic material. PV values as high as 100 000 psi = ft/min have been achieved, although in some cases there has been rapid wear. When filled with graphite, the wear rate and friction of the polyimide decrease considerably.

Table 9 shows some of the mechanical properties, coefficient of friction, wear factor, PV, and effect of temperature for the bearing materials described.

DESIGN CONSIDERATIONS

PV and wear.—The successful use of any non-lubricated 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.

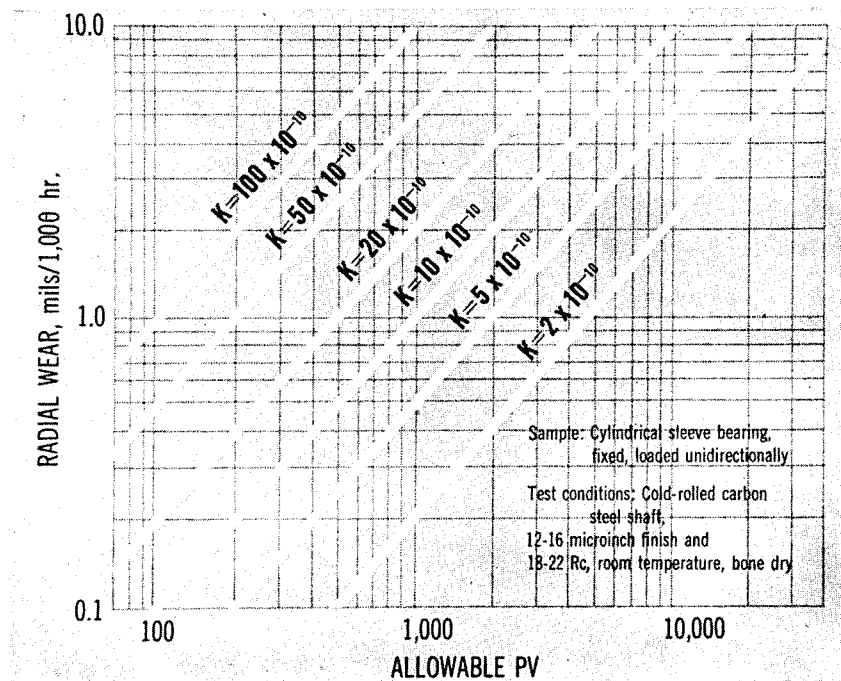


FIGURE 3.—Radial wear vs. PV as a function of wear factor.

The limiting PV can indicate whether or not a bearing material should be considered for a specific service.

Since wear is inevitable in non-lubricated rubbing contact bearing service, the designer 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:

$$R = KPTV$$

Figure 3, which is based on the equation, is a nomograph relating PV with unit wear per 1000 hr operation at various "wear factor" levels. Knowing the allowable wear of a given application, and the wear factor of a particular bearing material, the designer can determine an allowable PV. Conversely, having established a required PV level, unit wear can be reasonably predicted.

Clearances.—Good plastic journal bearing design requires the specification of proper running clearances on the shaft. Because of the high thermal expansion of most plastics, these bearings require larger clearances than those normally used with metal bearings. In some cases,

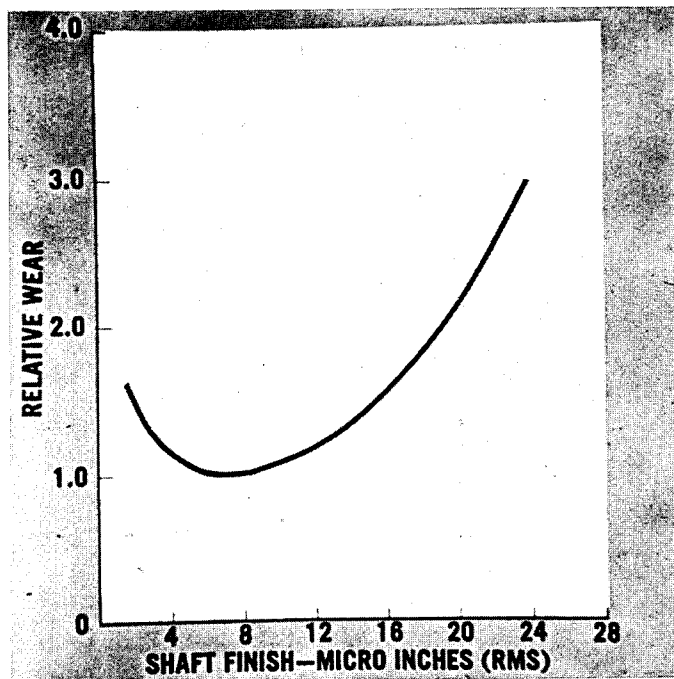


FIGURE 4.—Effect of shaft finish on wear of Teflon bearings.

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 diameter 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 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 μ in. RMS for best performance. Too smooth a surface is unnecessarily expensive and rougher surfaces tend to abrade the bearing. Figure 4 shows the effect of shaft finish on the wear of TFE bearings.

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



FIGURE 5.—Spherical bearings lined with fabrics of Teflon fluorocarbon fiber.

of the finished part cost. 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 which 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. Figures 5 through 15 show some typical applications.

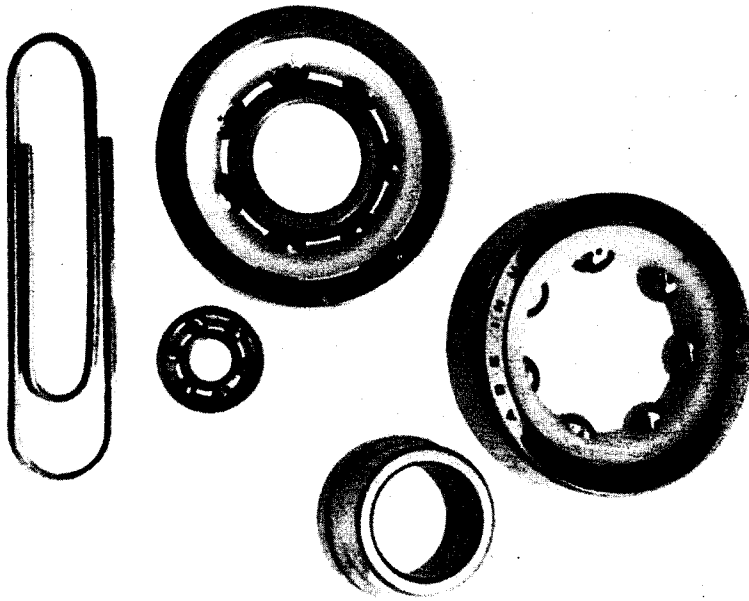


FIGURE 6.—Bearing retainers of delrin acetal resin.

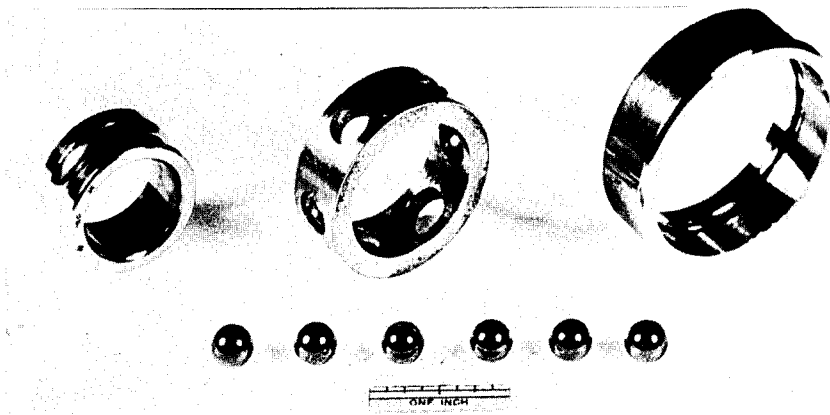


FIGURE 7.—Ball bearing retainers of filled Teflon TFE resin.

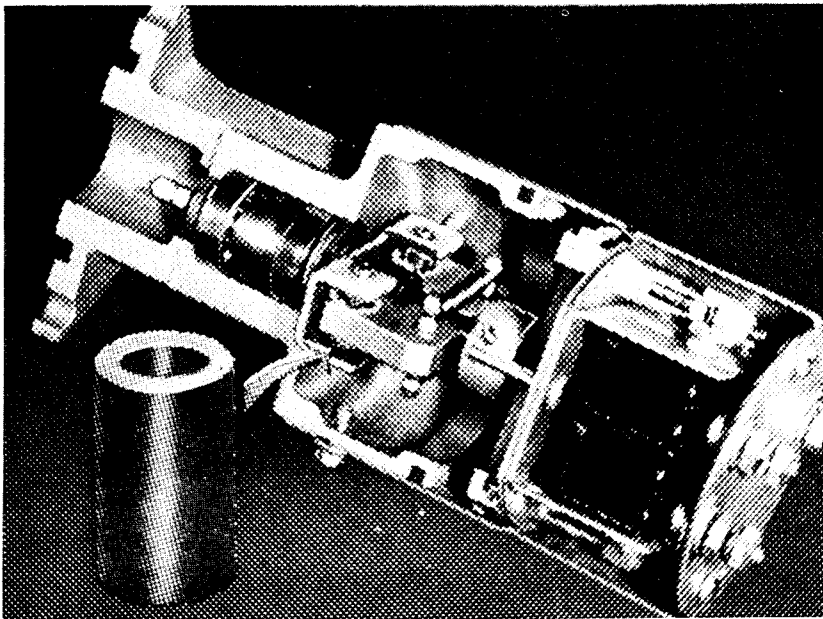


FIGURE 8.—Bearings of duPont Teflon TFE resins.

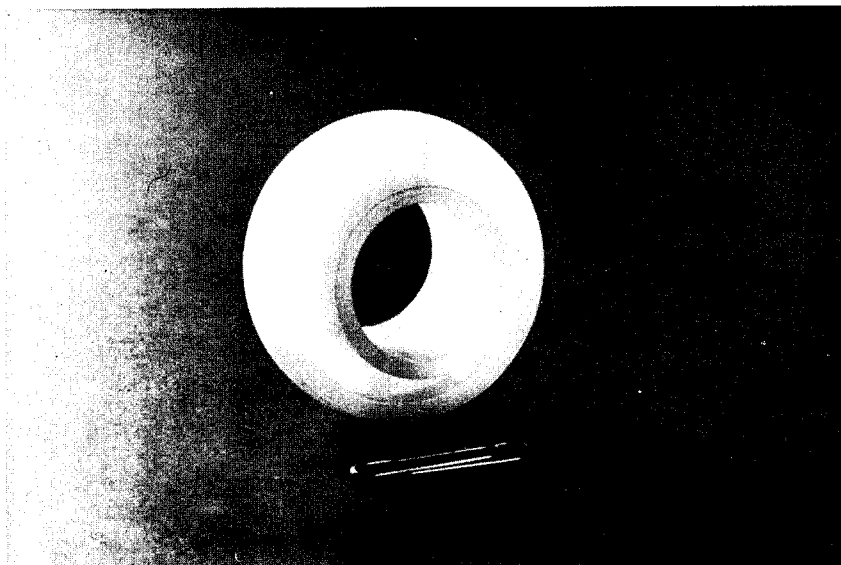


FIGURE 9.—Self-aligning bearing of zytel nylon resin.

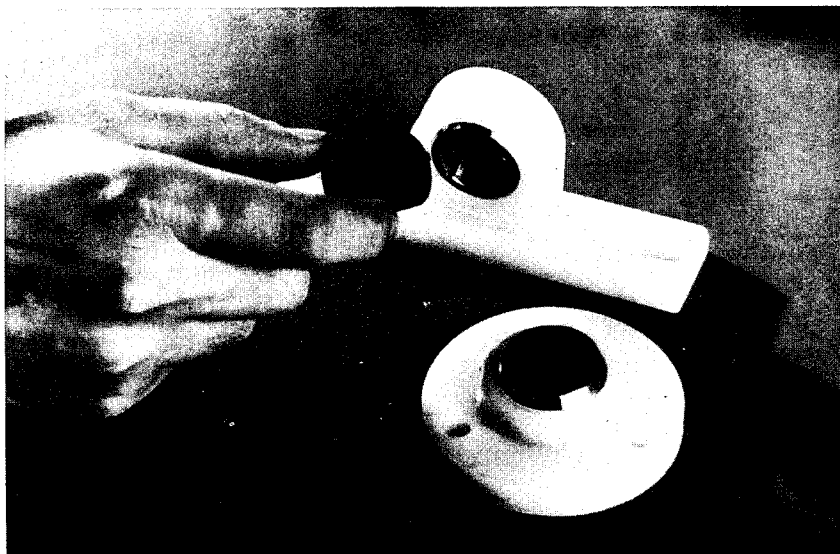


FIGURE 10.—Self-aligning bearing and housing of duPont delrin AF fiber/resin and zytel nylon resin, respectively.

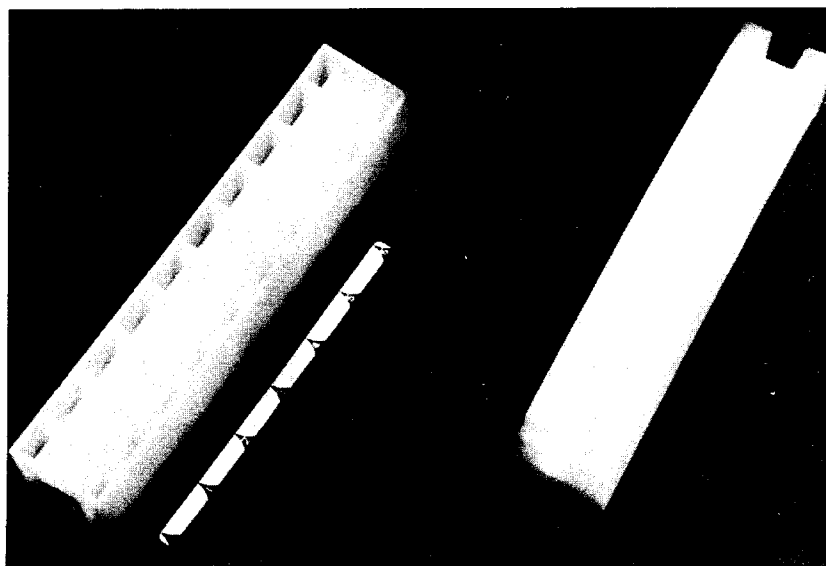


FIGURE 11.—Elevator gibs of duPont zytel nylon resin.

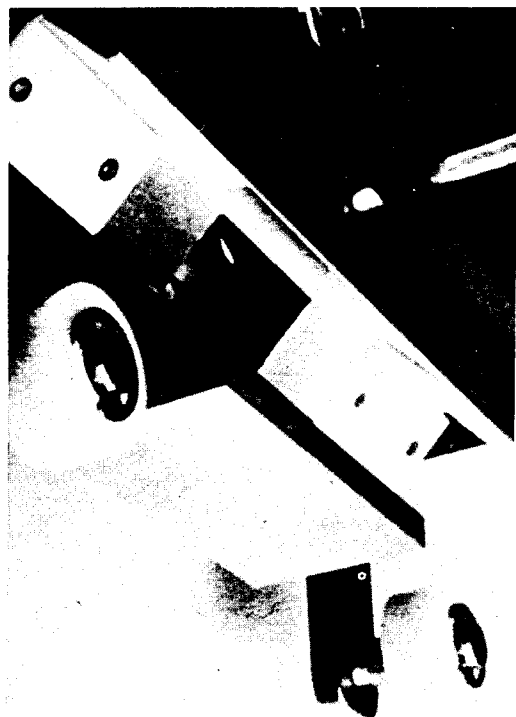


FIGURE 12.—Glide pads of Teflon resin.

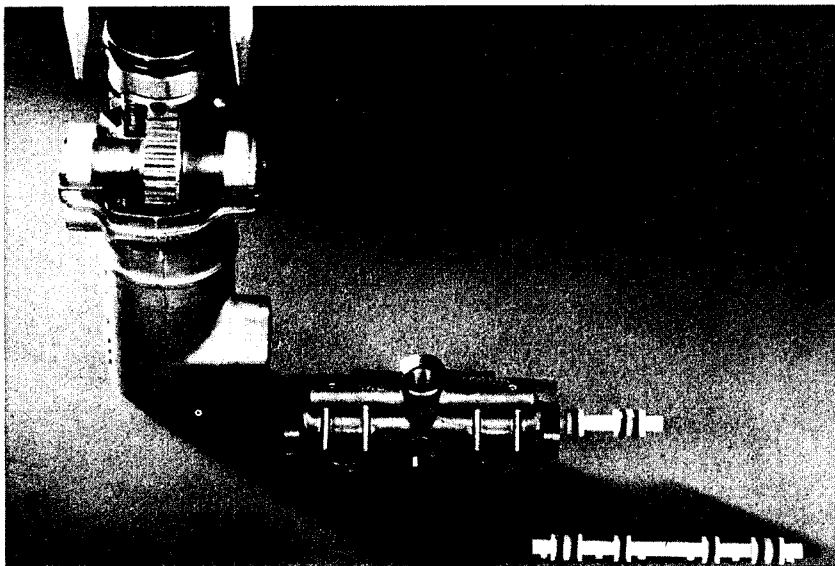


FIGURE 13.—Self-aligning bearings of zytel nylon resin.

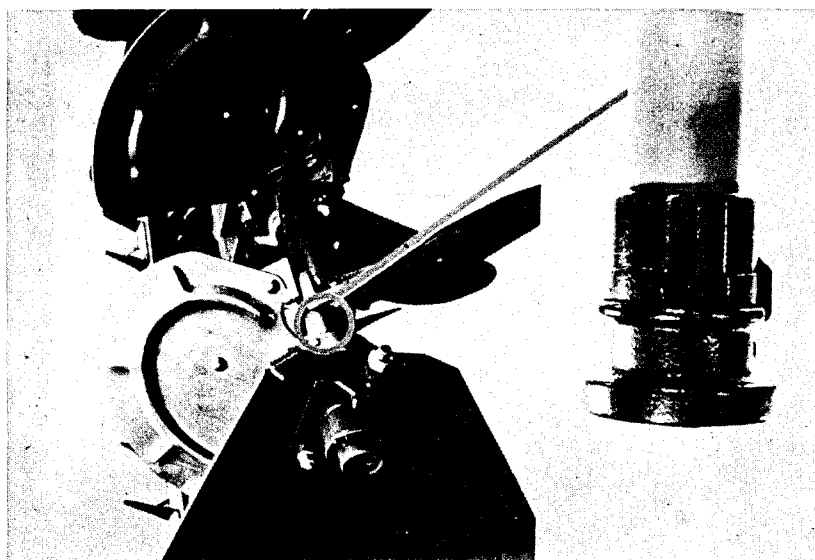


FIGURE 14.—Manure spreader bushings of duPont Teflon TFE resin.

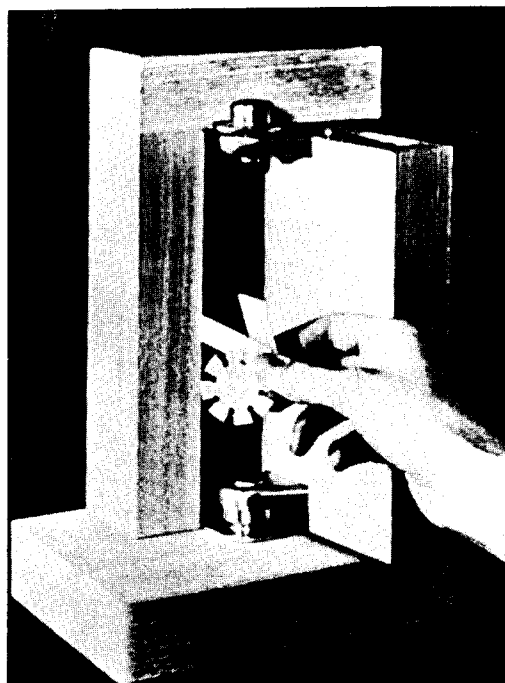


FIGURE 15.—Door pivot bearings of glass-filled tape of duPont Teflon TFE resin.

POWDERED SOLID LUBRICANTS

The simplest and oldest form of solid lubrication, still widely used, merely involves applying lubricating solids to the surfaces requiring lubrication. The types of powdered solids that can be used cover a wide spectrum of materials. The lubricating film is formed on the bearing or wearing surface by merely contaminating the surface with a finely divided lubricant powder. Rubbing tends to burnish the material and gives a better bond to the surface. 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 of application. This method 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.—No attempt is made to distribute the powder evenly. This method results in an uneven and generally unsatisfactory coating.

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

Aerosol spraying.—Finely divided lubricant is applied using an aerosol spray with a volatile carrier such as alcohol. Again, adhesion is not good; however, the ease of application may offset this disadvantage.

Examples of solid lubricant powders that can be applied using the above methods of application are molybdenum disulfide, graphite, mica, talc, and polytetrafluoroethylene. Applications are found in die lubricants, forming, press fitting, wire drawing, and fasteners (threaded and rivet).

The use of the powdered materials is usually limited to areas where adhesion of the lubricant is not critical. Many of the application techniques discussed in previous chapters were developed as a direct result of attempts to obtain better bonding of the materials in 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 of the inert variety (argon, nitrogen, etc.), 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; it has not been used in any known system.

Several investigators (refs. 1 to 4) have conducted studies to determine the feasibility of lubricating high-speed bearings by this technique.

A problem that can be anticipated with this method of lubrication is bearing fatigue. The phenomenon has been studied to some extent by Carter (ref. 5) using MoS_2 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. Lubrication is accomplished by a transfer of solid lubricant from within the structure of the composite to the interface between the moving parts. This transfer results in the formation of an effective lubricating film.

In 1950, personnel at the Lewis Research Center conducted an investigation to determine the feasibility of lubricating bearings in this manner. They also conducted an investigation to establish the amount of lubricant necessary to such a bearing material. Their studies (ref. 6) indicate that they investigated a material composition containing MoS_2 , up to 35 percent silver and 5 percent copper. They reported that effective lubrication could be obtained with a material containing 10 percent MoS_2 , and that this material (10 percent MoS_2 , 5 percent copper, 85 percent silver) 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 lubricating powder.

Other investigators have used this method of lubricating ball and roller bearings. Campbell and Van Wyk (ref. 7) report a lengthy study of lubricant composite development in which they were attempting to lubricate 204 size bearings operating in both air and vacuum 10^{-6} torr at 1500°F . Their report mentions between 400 and 500 different material combinations that were fabricated during their program. Unlike the Lewis workers mentioned earlier, they used extremely high percentages of lubricating powders (MoS_2 , PbO , etc.) and hard metal binders rather than silver. In this work their two best materials were reported to be 90 percent MoS_2 , 8 percent Fe, 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. Bowen et al. (refs. 8-11) of the Westinghouse Electric Corporation also have done considerable development work on the lubricant composite type materials. They have included such lubricant powders as polytetrafluoroethylene, graphite, antimony sulfide (Sb_2S_3),

iron sulfide (FeS), selenides and tellurides of tungsten, molybdenum, and many others. Work on these programs has been mainly on the lubrication of gears and related equipment.

Others, including Devine (ref. 12), have compacted the common solid film lubricants into solid sections. Personnel at Goddard and Marshall Space Flight Centers (refs. 13-15) and F. J. Clauss at Lockheed (ref. 16) have investigated electrical brush materials comprised of metal and lubricant powders.

Method of Evaluating Solid Film Lubricants

Methods of evaluating solid film dry lubricants are almost as numerous as the lubricants themselves. 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 these materials over the range of conditions that he feels is important to him. Along with the wear life and friction type tests, there are other tests which are nearly standard throughout industry. These minor tests include film adhesion, fluid resistance, heat resistance, and corrosion resistance. Nearly all 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. The tests also showed that very little repeatability existed between the same machine in different laboratories. They did find that more repeatability existed between laboratories using the Falex tester as opposed to 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.

M. J. Devine (ref. 1) has compiled a list of test machines being used today in the evaluation of solid lubricants. Table 10 is a reproduction of this list.

Most of the test data on solid dry lubricants are concerned with 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 development tools (Chapter 3, refs. 1, 2 and 3). Because no single organization has attempted to design and

TABLE 10.—*Examples of Friction and Wear Test Apparatus for Solid Lubricants*

TEST APPARATUS	CONDITIONS	SPECIMEN
Plain Bearing Tester	1. 630F 2. 10 cpm 3. $\pm 32^\circ$ /cycle 4. 10,000 psi	H-11 Test Shaft, M-2 Steel Bushing
Friction Tester	1. 77F 2. 65 cm/sec 3. Variable load	Metal disk and rider
High Temperature, High Speed Bearing Performance Apparatus	1. 750F 2. 10,000 rpm 3. 5 lb Thrust Load, 3 lb Radial Load	20 mm rolling contact bearing
Vacuum Friction and Wear Apparatus	1. 77F 2. 390 fpm 3. 1000 g 4. 10^{-6} to 10^{-7} mm Hg	Metal Disk and Rider
Lubricant Vacuum Environment Apparatus	1. 77 to 1000F 2. 1250 rpm 3. 5 lb Thrust Load 3 lb Radial Load 4. 1×10^{-6} mm Hg	20 mm rolling contact bearing
Modified 4-Ball Wear Machine	1. 1000F 2. 200-700 ft/min 3. 2-8 Kg	$\frac{1}{2}$ " diam balls
High-Temperature, High Speed Rubbing Wear Apparatus Pellet Friction Machine	1. 1000-1800F 2. 100-200 ft/sec 3. 5-50 psi 1. 80-1000F 2. 600 ft/min 3. 128-326	Rotating Washer vs. stationary pad stators Pellet vs. tool Steel Track
Press Fit Tester	1. 77F 2. 0.6-2 in/min 3. Variable Load	Pin and Bushing
Lubricant Tester Model LFW-I	1. 77F 2. 12-197 rpm 3. 30-630 lb	Test block and $1\frac{3}{8}$ " timken bearing outer race
Falex Lubricant Tester	1. 77F 2. 290 rpm 3. 200-2500 lb (gauge)	Cylindrical pin and V-blocks
Timken Tester	1. 77F 2. 200-1300 rpm 3. 6300-50,400 psi	Cylindrical cup rotating against a stationary rectangular block Rotating ring vs. stationary blocks
Oscillating Tester	1. 400F 2. 230 cycles/min 3. 62 deg. arc 4. 700 lb	Cylindrical pin and V-blocks
Dry Film Lubricant Tester	1. 100-1700F 2. 3.5 ft/min 3. 300 lb	Cylindrical sleeve slider sliding on a cylindrical rod
Sliding Friction Test Apparatus	1. 77F to 300F 2. 47.0 slides/hr 3. Oscillated $\pm 48^\circ$ 4. 10^{-6} - 10^{-9} torr	Cylindrical sleeve slider sliding on a cylindrical rod
Bearing Test Apparatus	1. 77F 2. 1800 rpm 3. $\frac{3}{4}$ lb axial and $\frac{1}{2}$ lb radial 4. 10^{-7} - 10^{-8} torr	20 mm rolling contact bearing
Fretting Corrosion Apparatus High Temperature Bearing Radial and Thrust Rig	1. 77F 1. 1200F 2. 20,000-35,000 rpm 3. 50 lb thrust and/or 50 lb radial	Steel flat vibrating against steel flat 20 mm rolling contact bearing

TABLE 10.—(Concluded)

TEST APPARATUS	CONDITIONS	SPECIMEN
Lubricant Tester (Modified)	1. 77F 2. 72 rpm 3. 630 lb	Rotating ring vs. Rectangular block
Modified Falex Tester	1. -196C (liquid N ₂) 2. 290 rpm 3. 1000 lb (gauge)	Rotating pin vs. Stationary V-blocks
Crossed Cylinders Friction Apparatus	1. 77F 2. 8-367 sfm 3. 1-5 lb	Vertical cylinder-carbon tool steel, horizontal cylinder-solid section
Model A-3 Friction and Wear Tester	1. 77F 2. 72 rpm 3. 630 lb	Timken test cup T-54148 and AISI- 4130 steel blocks
Stick-Slip Slow- Speed Friction Apparatus	1. 80-1000F 2. 0.17 mm/sec 3. 64,000-100,000 psi	1/2" diam ball vs. flat block
Rolling and Sliding Contact Specimen Test Machine	1. -100 to +2000F 2. 22 cpm 3. duration-30 min	Ball and plate
A-6 Friction and Wear Tester	1. -60 to 1500F 2. 200 fpm 3. 16000 psi	Rotating ring vs. rectangular blocks
Galling Machine	1. 900F 2. 3.5-300 fpm 3. 20-2500 psi	Flat plate vs. test cup

construct a standard type test machine for dry lubricants, the industry has relied heavily on equipment used to evaluate liquid lubricants. The lack of standard methods of evaluation has created problems in comparing the results of various programs, but many of the solid lubricants do not lend themselves to standardization of test methods.

FRICITION AND WEAR TESTERS

Although table 10 lists a wide variety of friction and wear tests that have been developed for the evaluation of solid lubricants, only the most common machines will be discussed in this chapter, and are listed in table 11.

FALEX LUBRICANT TESTER

Since the organizations controlling the dry lubricant specifications are using the Falex test machine exclusively in their test work, this machine will be considered first. A Falex test fixture is shown in figure 16. It consists of a rotating pin held between two stationary V-blocks in a nutcracker-type loading system. 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 (ref. 2), it has been modified for testing at both liquid nitrogen and elevated temperatures (refs. 3, 4). A more complete description of test machine and procedure is given in reference 5.

Another extensively used test configuration 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 17. Figure 18 shows a similar

TABLE 11.—*Common Evaluation Testers for Bonded Solid Lubricants*

Name	Specimen geometry	Temperature (° F)	Environment	Load	Ref.
Falex Lubricant Tester	Pin between two V-blocks (see fig. 2)	Ambient	Air	Variable to 4500 lb	2
Alpha Molykote LFW-1	One block on rotating disk (see fig. 5)	Ambient	Air	Variable to 600 lb	5
Alpha Molykote LFW-2	One block on rotating ring (see fig. 8)	Ambient	Air	Variable to 1100 lb	6
Hohman A-6	Two blocks on rotating disk (see fig. 4)	Ambient to 1600°	Air	Variable to at least 600 lb	
Midwest Research Institute Mark VB	Two blocks on rotating disk (see fig. 4)	Ambient to 1500°	10 ⁻⁶ torr	Variable to 600 lb	
MRI Three Pellet Wear Life Apparatus		Room Temp.	Air on N ₂	300 gm/ contact	
MRI Vacuum Friction Apparatus	Three pellets on flat track (see fig. 16)	Ambient to 400°	10 ⁻⁷ torr	300 gm/contact	8
MRI Oscillatory Bearing Tester	Plain spherical bearing* (see fig. 19)	Room Temp. to 1000°	Air	Variable to 10 000 psi	
Alpha Molykote LFW-3	Annular ring on flat block (see fig. 21)	Ambient to 1200°	Air	Variable 100-5000 lb.	9
Alpha Molykote LFW-4	Pin and bushing (see fig. 23)	Ambient	Air	Variable to 12 000 lb	10
NASA High Load Machine	Flat on flat (see fig. 27)	Room	Air	Variable to 150 000 psi	11
NASA Spherical Rider	3/16 in. hemispherical rider on rotating flat disk (see fig. 29)	Variable Cryogenic to 2000°	Air	Variable	12
(Westinghouse) Friction Tester	One rotating ring on a flat disk	Ambient	Air	Variable to 2000 psi	14

* Bearing locked to shaft to cause rotation on spherical surface.

arrangement, 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.

Brief descriptions of several machines in this category follow:

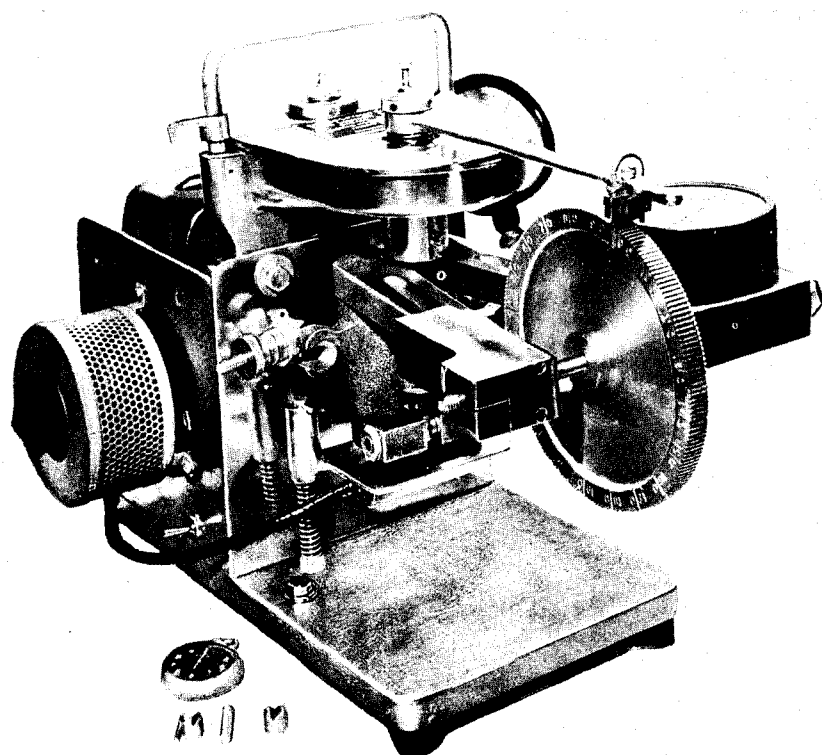
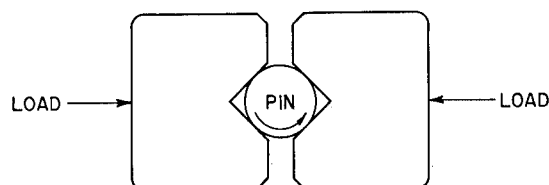


FIGURE 16.—Falex lubricant tester.

LFW-1

This machine uses the test configuration shown in figure 19, where a stationary test block is pressed against a rotating Timken* ring. The rotational speed is variable from 12.5–197 rpm. Load is variable from 30–630 lbs. Testing is at room temperature only, and friction can be monitored through the test period. The machine can be operated in both unidirectional and oscillatory motion. This test machine gives initial line contact, with area contact resulting from load and wear. Figure 19 shows the test machine and related equipment, while figure 20 shows the test area.

* Timken—Registered Trade Mark.

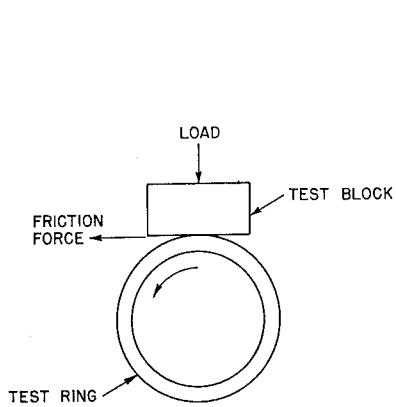


FIGURE 17.—Single rub shoe on a rotating ring.

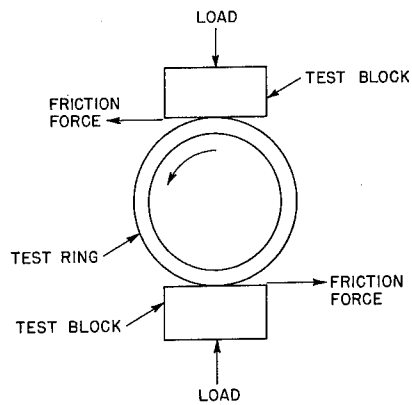


FIGURE 18.—Diametrically opposed rub shoes on a rotating ring.

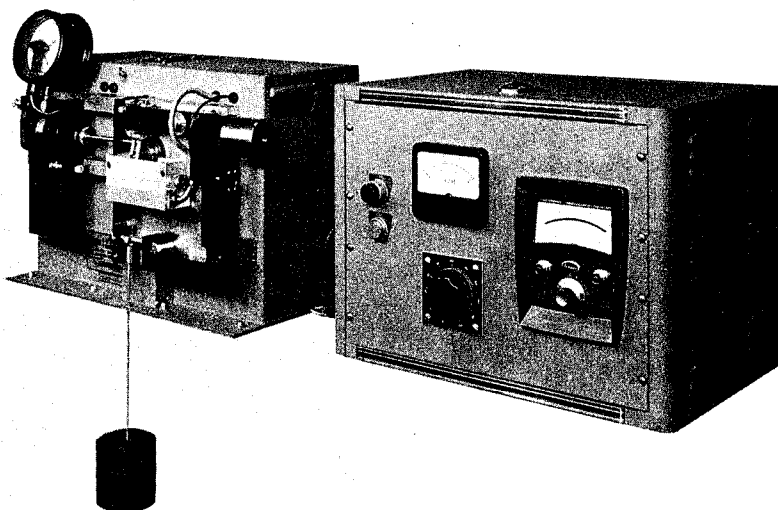
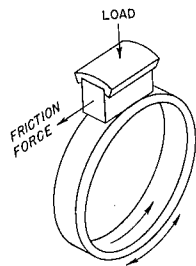


FIGURE 19.—LFW-1 test machine.

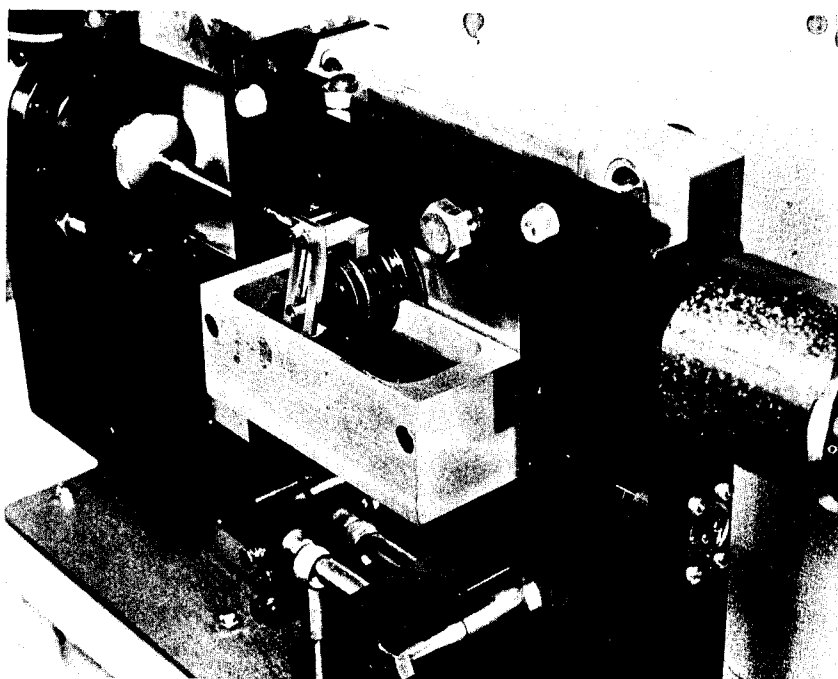


FIGURE 20.—LFW-1 test area.

LFW-2

This machine is a refined Timken tester utilizing the test configuration shown in figure 21. Rotational speed is variable from 0–1100 rpm. Load is variable from 0–1000 lbs. Friction, wear, and frictional temperature can be obtained under varying speed and load conditions. The machine gives initial line contact with area contact resulting from load and wear. The machine is shown in figure 21, with additional information in reference (6).

HOMAN A-6

The Hohman A-6 tester utilizes the diametrically opposed blocks shown in figure 18. Load is variable to 600 lbs. The machine is capable of operation to 1600° F. As with the above two machines, initial line contact is obtained with area contact resulting from wear and load. A schematic drawing of the test configuration is shown in figure 22. A Hohman A-6 tester is shown in figure 23.

MIDWEST RESEARCH INSTITUTE 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 showing the

*Hohman Plating and Mfg., Inc., Dayton, Ohio.

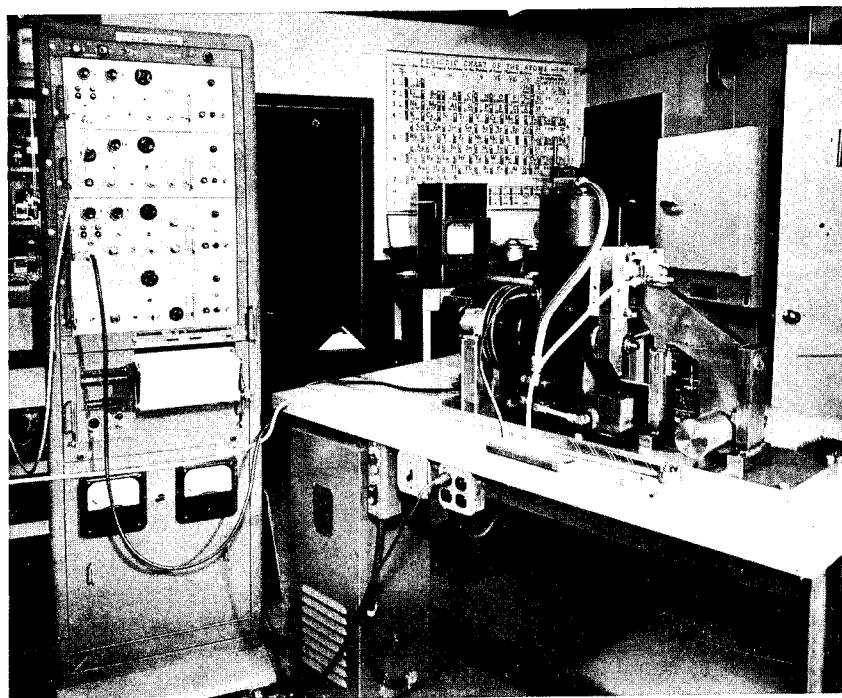
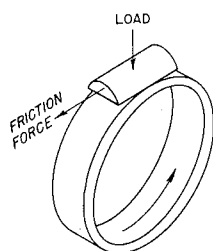


FIGURE 21.—LFW-2 test machine.

test configuration and loading system is shown in figure 24. Capabilities of this machine are: 600 lbs on each shoe, variable speed 175–3500 rpm (continuously variable), room temperature to 1500° F, and vacuum of 10^{-6} torr. The machine is shown in figure 25. A more complete description can be found in reference (7).

MIDWEST RESEARCH INSTITUTE—PELLET WEAR LIFE APPARATUS

This small type tester was developed by MRI for NASA's Marshall Space Flight Center. A schematic drawing and photograph are shown

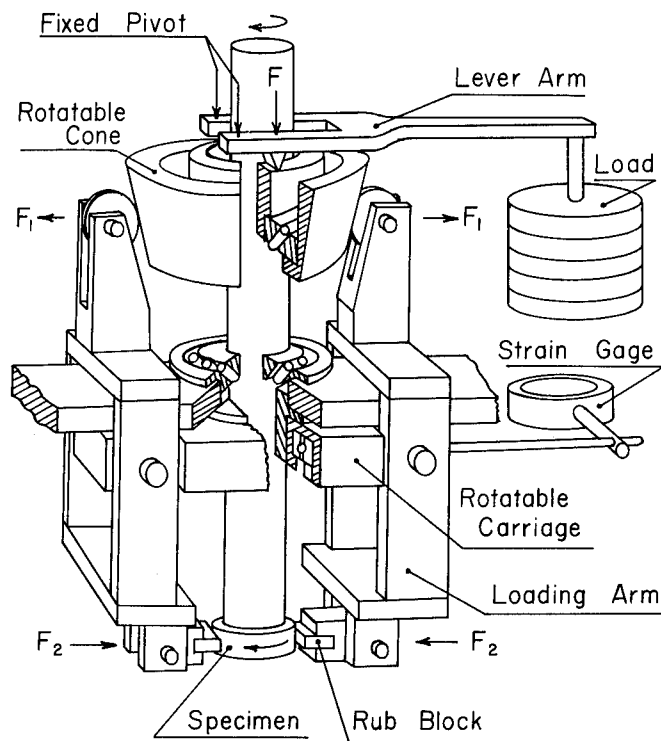


FIGURE 22.—Hohman A-6 test configuration.

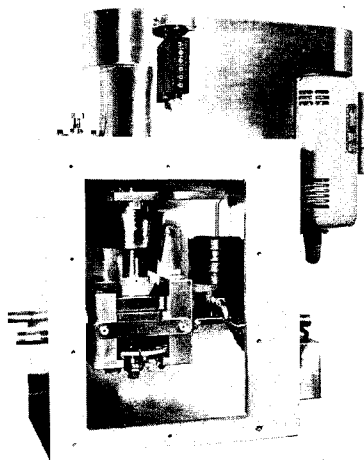


FIGURE 23.—Hohman A-6 test machine.

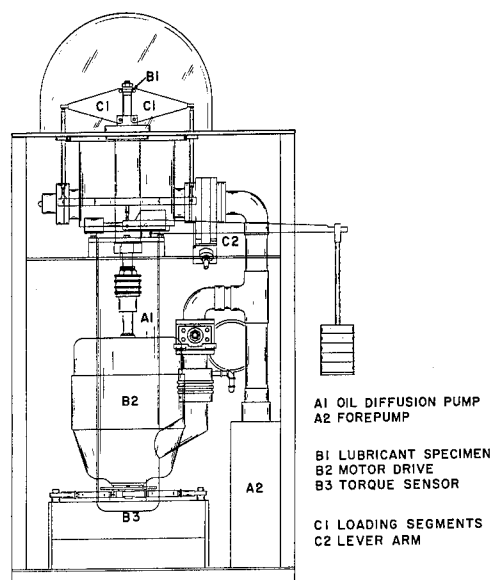


FIGURE 24.—MRI Mark VB test configuration and loading system.

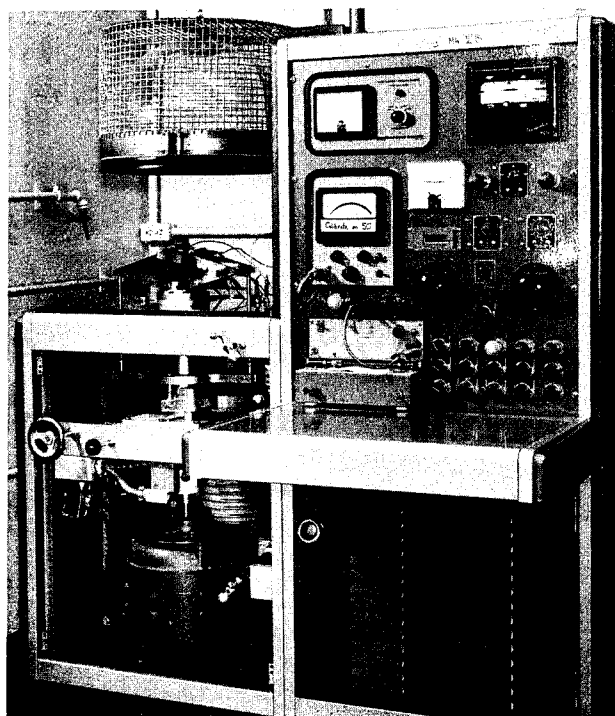


FIGURE 25.—MRI Mark VB test machine.

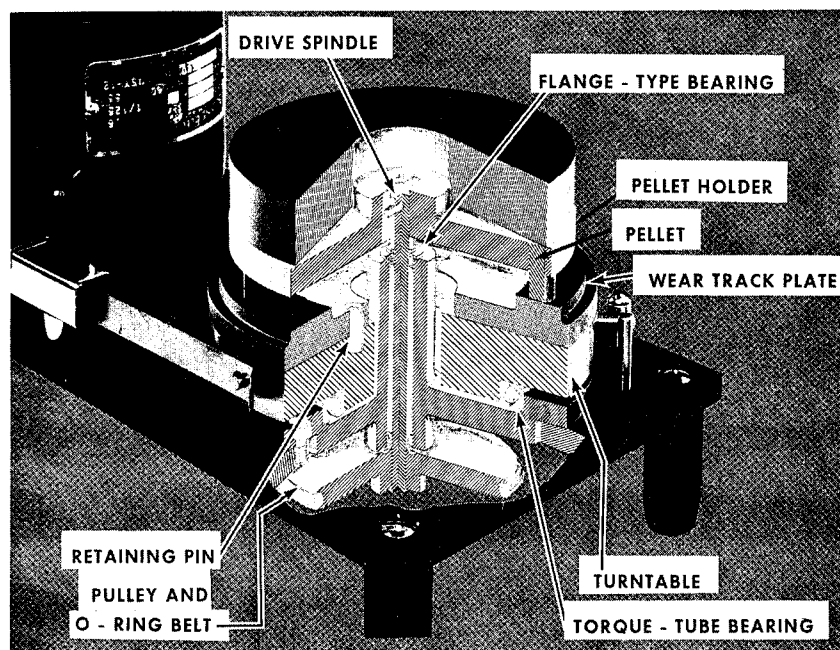


FIGURE 26.—MRI wear life tester (cut-away).

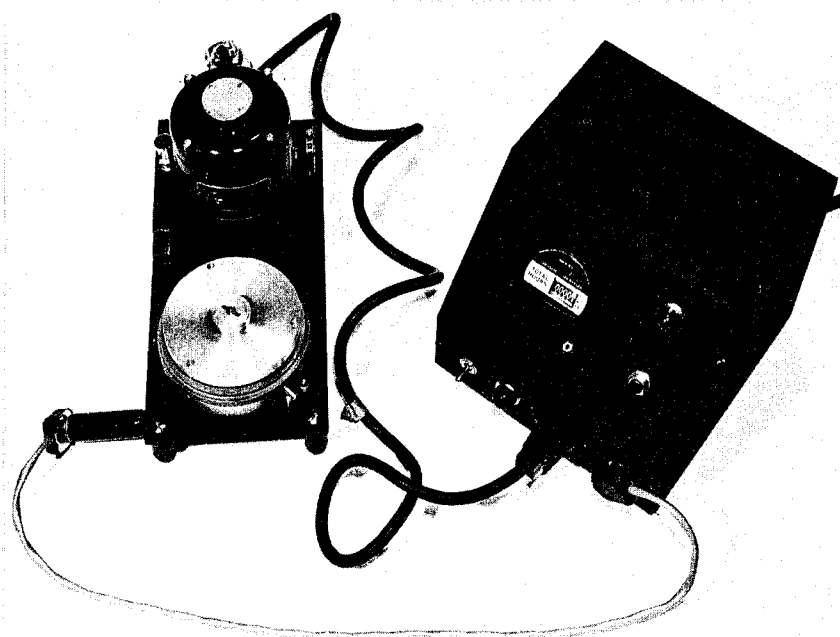


FIGURE 27.—MRI wear life test machine.

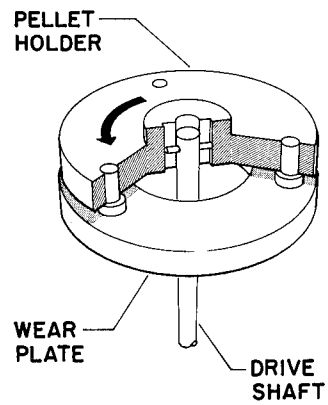


FIGURE 28.—MRI wear life test configuration.

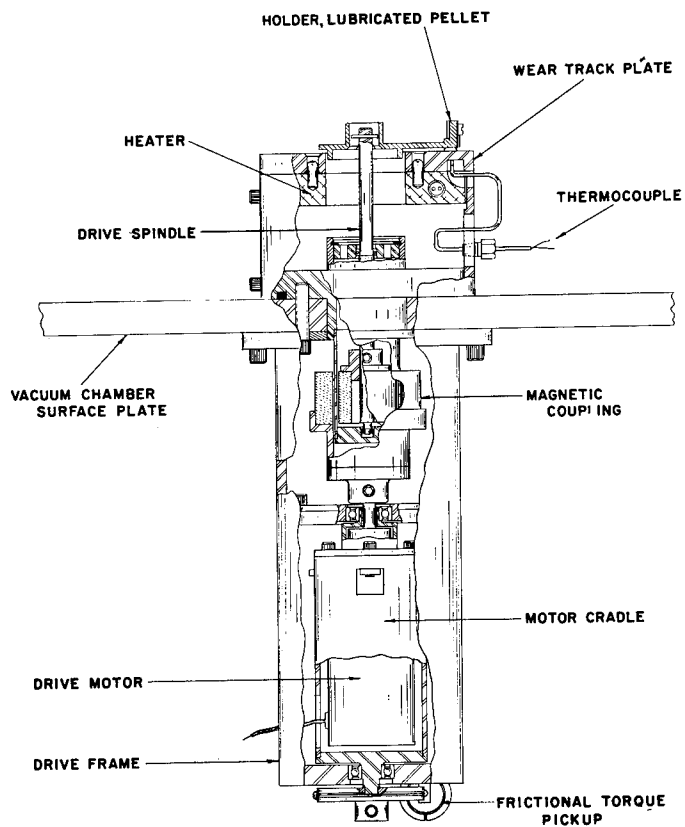


FIGURE 29.—MRI vacuum friction apparatus drive assembly.

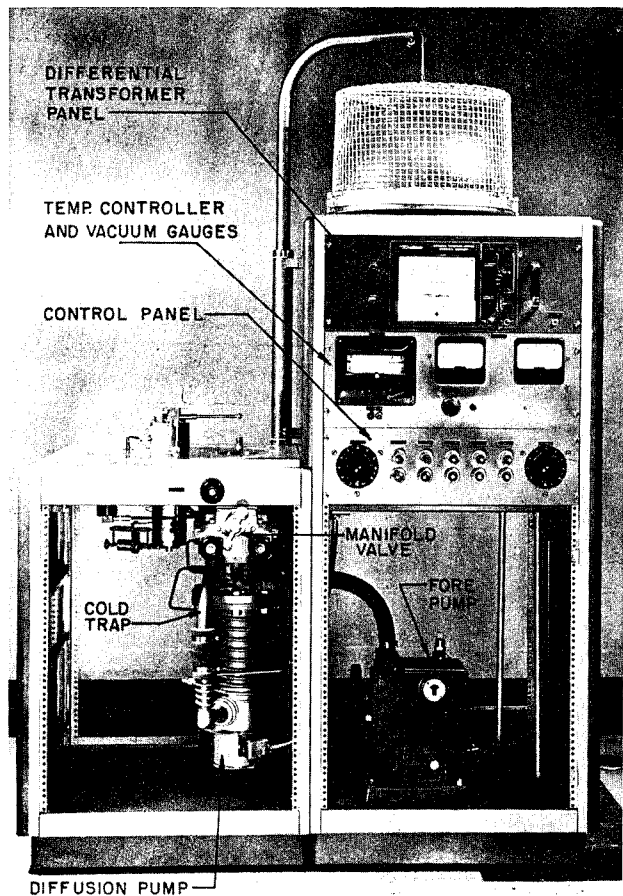


FIGURE 30.—MRI vacuum friction apparatus.

in figures 26, 27, and 28. This apparatus is intended for use in conducting, primarily, wear-life investigations at light loads. It is designed to operate unattended for extended periods of time. The specimen configuration is three $\frac{1}{4}$ -in. diameter pellets sliding over a flat plate. The unit has the following capabilities: load 300 grams/contact, speed to 765 fpm, room temperature, and normal atmosphere.

MIDWEST RESEARCH INSTITUTE—VACUUM FRICTION APPARATUS

This equipment uses the same specimen configuration shown in figure 28. However, this equipment is designed for operation at a vacuum of 10^{-7} torr. Figure 29 shows the drive mechanism for this equipment, and figure 30 shows the entire machine. Additional information is available on these machines from reference (8).

MIDWEST RESEARCH INSTITUTE—OSCILLATORY BEARING TESTER

Machine capabilities are 10 000 lb load, room temperature to 1000° F, oscillatory motion of ± 15 at the rate of 9 cycles per minute. Test specimens are $\frac{5}{8}$ -in. bore plain spherical bearings. Tester and specimen configurations are shown in figures 31 and 32. On this tester, the spherical bearing is locked on the shaft causing the bearing to operate on the spherical area.

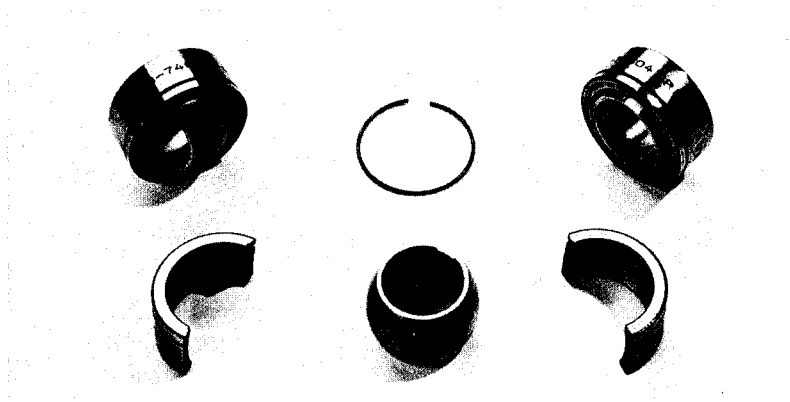


FIGURE 31.—Spherical bearing for solid lubrication, $\frac{5}{8}$ -in. bore.

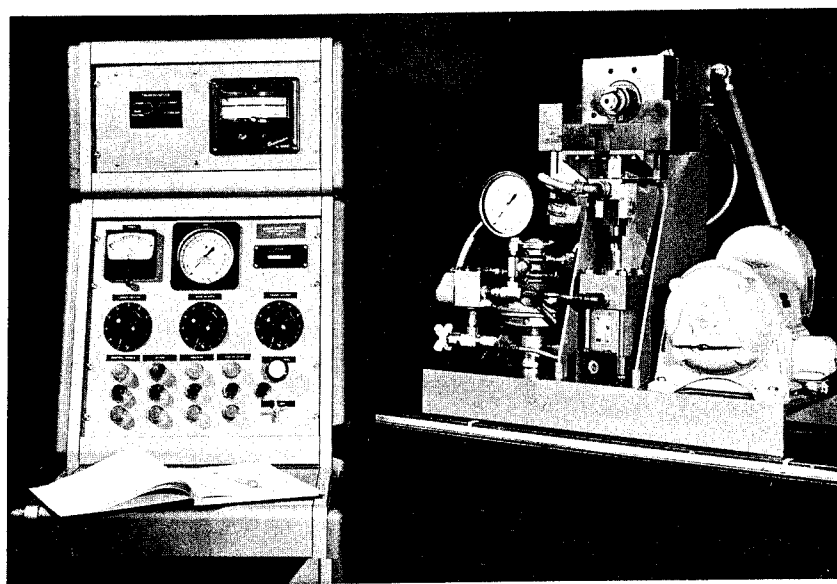


FIGURE 32.—MRI oscillatory bearing test machine.

LFW-3

Machine capabilities are: room temperature to 1200° F, 100–5000 lb load, rotational speed to 9–320 rpm, and oscillatory frequency of 6–227 cycles per minute (0–60°, 6–227 cpm; 0–120°, 6–120 cpm). Test specimens are of the annular ring variety with a constant theoretical area of contact as shown in figure 33. Additional information is available from reference (9).

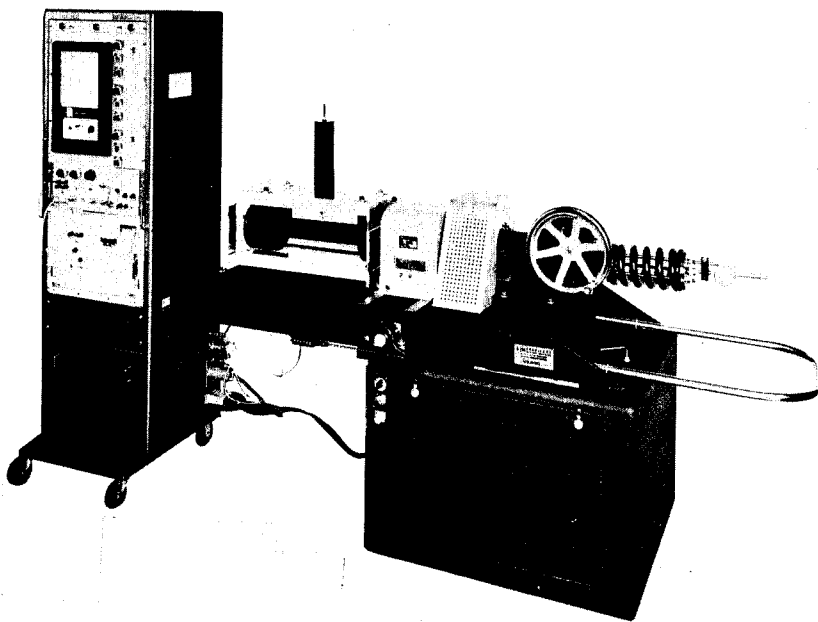
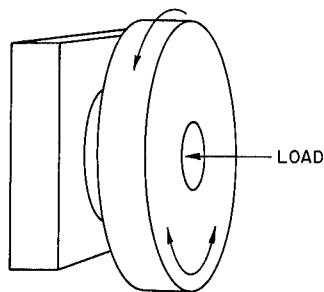


FIGURE 33.—LFW-3 test machine.

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 34. The machine is intended for studies on static and kinetic friction during and after "wear-in" galling, seizing, and the problem of stick-slip. Figure 34 shows the entire machine. Additional information can be obtained from reference (10).

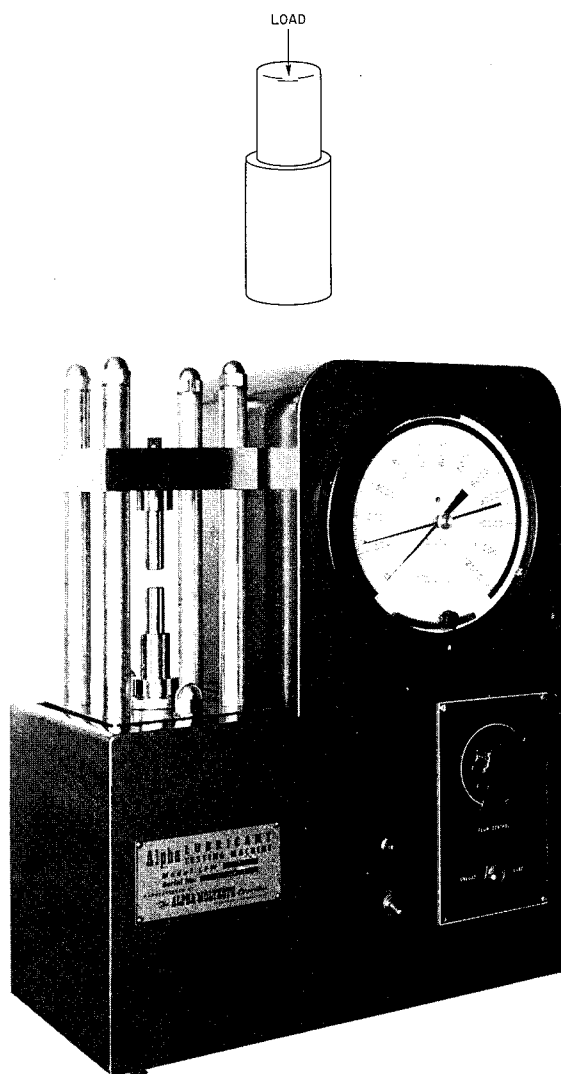


FIGURE 34.—LFW-4 test machine.

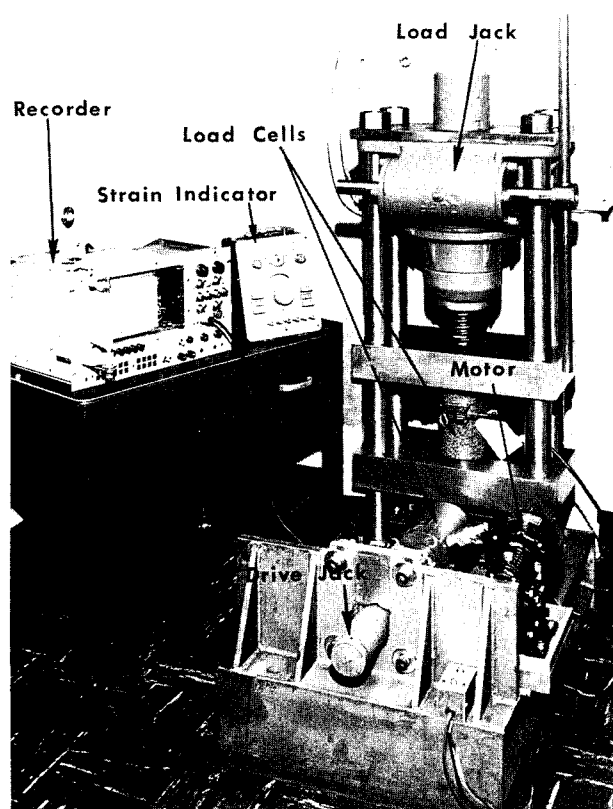


FIGURE 35.—NASA (Marshall) high load test machine.

NASA—HIGH LOAD TESTER (MARSHALL SPACE FLIGHT CENTER)

A photograph of this test device is shown in figure 35 and a schematic in figure 36. The test machine consists of a frame on which is mounted a 50-ton mechanical jack. The jack 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 36 is positioned between the self-aligning plates and is connected through a load cell to the drive mechanism. The machine is capable of unit loads of 150 000 psi on projected areas of 0.59 sq in. Speed is low, 0.67 in./min, with total travel of $\frac{1}{2}$ in. in each direction. Additional information is available from reference (11)

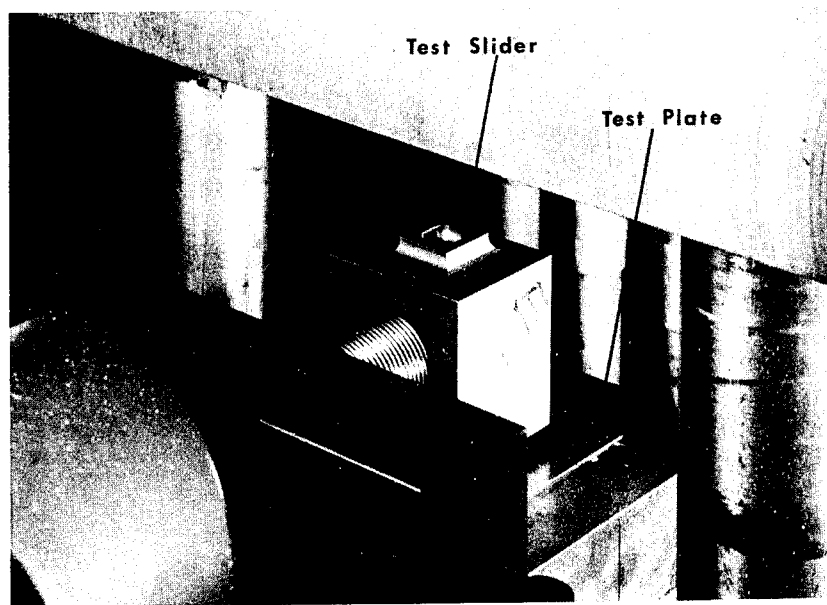
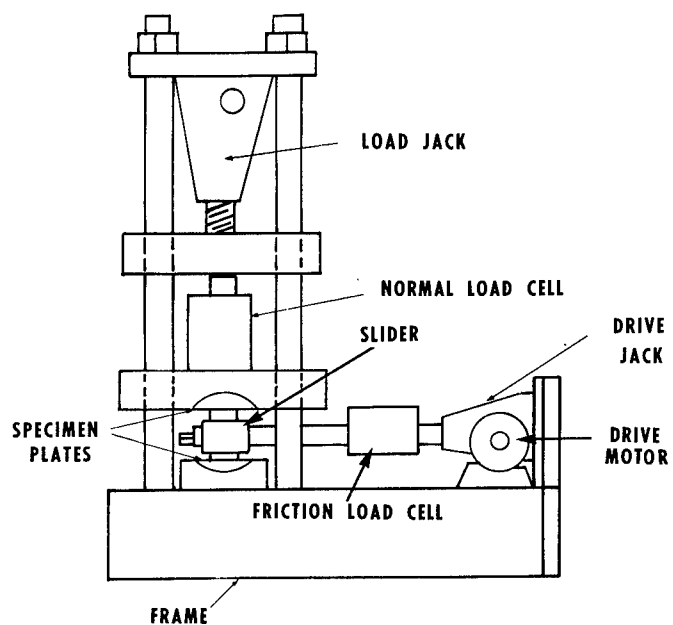


FIGURE 36.—NASA (Marshall) high load tester, (a) schematic, and (b) slider block.

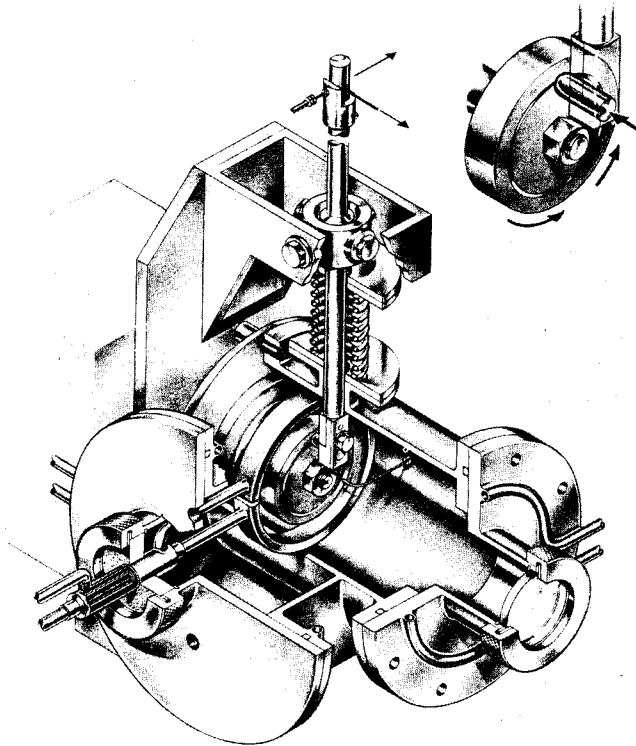


FIGURE 37.—NASA (Lewis) friction and wear test machine.

NASA—FRICTION AND WEAR MACHINE (LEWIS RESEARCH CENTER)

Figure 37 shows the arrangement of this machine and gives a close-up view 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 1000g 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 (12) and (13).

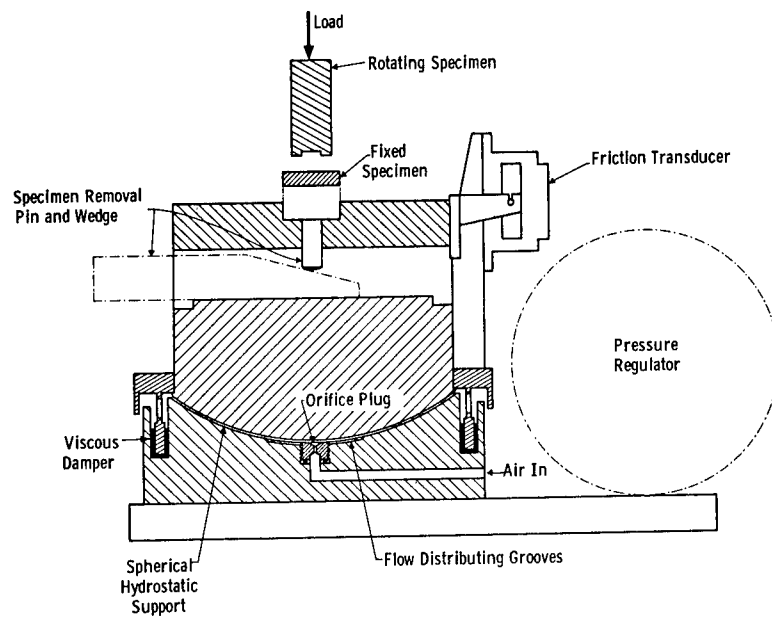
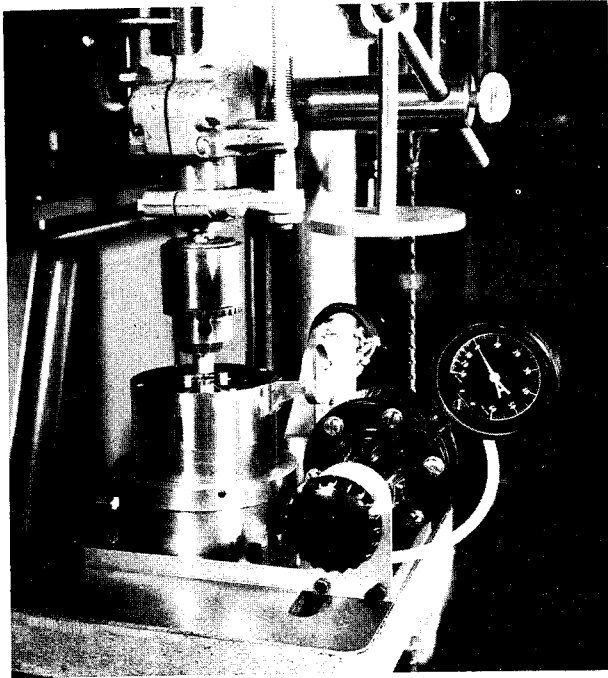


FIGURE 38.—Westinghouse friction tester.

WESTINGHOUSE FRICTION TESTER

This tester is a portable device expressly capable of accurate friction measurement of small $\frac{1}{2}$ -in. diameter specimen which can be run dry over a wide range of speeds and loads. The friction measuring accuracy is achieved by the use of a pneumatic self-aligning seat for the fixed disk specimen. The drive system can be a drill press. Figure 38 shows a cross-section view of both the test specimens and test device, with additional information in reference (14).

The North American Aviation plain bearing tester is briefly described by F. J. Williams in the Appendix. It is again emphasized that, although many testers are available for the testing of bonded solid lubricants, the results from any one tester cannot be taken as the absolute answer.

CHAPTER 7

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 types cannot tolerate. Other advantages over conventional lubricants include (a) lubrication for the life of the part, (b) lubrication without generation of detrimental wear debris, (c) essentially dry lubrication, (d) lubrication of parts of components that are difficult to reach, and (e) lubrication where the presence of vapors from fluid or grease lubricants cannot be tolerated.

Environmental effects can have strong interactions and specific performance may vary from device to device for the same lubricant. The reader 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 application. 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 so than other lubricants, requires careful consideration of its actual operating conditions.

NORMAL ATMOSPHERE

Of the large amount of research being conducted and the large amount of literature now available on solid lubricants, only a small amount deals with the behavior of solid lubricants in normal air atmosphere at temperatures in the range of -65° to $+200^{\circ}$ F. Nearly everyone is interested in very specific and extreme conditions. Apparently, the feeling is that since there are specifications covering the solid lubricants at

normal conditions, no further work needs to be done. No one has attempted to arrange the known characteristics of solid lubricants in normal air atmosphere into a usable design-type document.

In the normal atmosphere, the solid lubricants come into contact with such things as dust, dirt, water vapor, salt water, oil lubricants, and many others, all of which adversely affect their usefulness. A good example is water vapor, or humidity. Several investigators have shown that water vapor causes molybdenum disulfide solid lubricants to have higher coefficient of friction values, 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 corrosion. Water can enter the pores and react with the base material or substrate forming rust or corrosion. The situation is further aggravated in the presence of a salt solution. Personnel at the Rock Island Arsenal, (refs. 1, 2) have conducted extensive corrosion studies on resin-bonded solid lubricants. They report 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 report that conventional lubricants are harmful to the wear life of a bonded solid film lubricant. (This group was responsible for the development of the corrosion resistant lubricant R1A #9 described in Chapter 4.)

The MRI group has recently conducted a corrosion study using the MLF-9 lubricant mentioned in Chapter 4. Incorporation of metal chromates with the MLF-9 improved its corrosion resistance. However, over-coating the lubricant with the resin-bonded Teflon material provided much more corrosion protection.

The problem of corrosion encountered when using solid lubricants has been investigated by several other researchers (refs. 3-5). They also observed (ref. 5) 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 1 000 psi is *not* recommended.

For those interested in additional corrosion studies on solid lubricants, the Coordinating Research Council (CRC) is conducting a round-robin testing program. The results of these tests have not been released.

From conversations with many experts in the solid lubricant field, it appears 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 solubility of the silicate material. This problem has also been encountered when

attempting to test a sodium silicate bound material at liquid nitrogen temperatures. Apparently, the solubility problem encountered with the silicate materials can be overcome by an adequate curing process.

Since 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 MoS_2 -graphite films.

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, radiation zones, etc. Many of the solid lubricants will not provide lubrication in all of these various 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 deleterious to solid lubricants containing molybdenum disulfides. This is particularly true as the temperature increases, causing a rapid oxidation of the MoS_2 to MoO_3 . However, there are solid lubricant films that work just the opposite, one being a $\text{PbO-Pb}_3\text{O}_4$ film reported by Peterson and Johnson (refs. 6, 7).

The national space programs have aroused great interest in solid lubricants for the vacuum environments 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 such systems. 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 5, 6, 7, 8, and 9 reported in Chapter 5. The lubricating plastics, polytetrafluoroethylene and polyimide, are reported to be good lubricants for vacuum service. There are other hostile environments where solid lubricants can be considered for use, especially in the handling of reactive chemicals and gases. Lubricating plastics are finding many applications in these areas.

TEMPERATURE EFFECTS

The resistance of various solid lubricants to temperature extremes has been a major stimulant in the development of solid lubricant materials. Various types of solid lubricants perform well at temperatures in the cryogenic region and at temperatures up to 2000°F . Conventional

petroleum-based materials have upper limits of only 200° to 300° F. Advanced products and synthetic fluids have operating ranges from 600° 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° 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 anti-seize material on threaded applications, where very little oxygen is in contact with the material and rapid oxidation cannot take place. With the 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 temperature can be attributed to brittleness of the resin binder and at elevated temperatures to the decomposition of the organic resin binder. At both extremes film adhesion is very poor. It appears that the above condition 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 Chapter 3 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 (MoS_2 —graphite—sodium silicate) are designed for operation over a wider temperature extreme than are the resin-bonded materials. They are reported to be usable from -300° to 750° F in air and to in excess of 1200° F in vacuum (ref. 8). With these films, the lubricating solid (MoS_2) limits its use for continuous operation above 750° F, because it rapidly oxidizes to MoO_3 , an abrasive type material.

An interesting observation made by Gaddis (ref. 9) is that the friction of the sodium silicate bound solid lubricants decreases with increases in temperature up to about 350° F and then begins to increase slightly. Another very desirable characteristic of these films is their compatibility with liquid oxygen material, which makes them prime candidates for use on space vehicles.

The ceramic-bonded films (CaF_2 —ceramic binder) are designed for use in the temperature range of 1000° to 2000° F, although they have been shown to be usable at much lower temperatures (as discussed in Chapter 4). At ambient temperatures below 700° F, these films show reduced performance and, therefore, should not be considered as low temperature lubricants. However, if the ceramic-bonded films are used at low temperatures (< 700° 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 melting of the binder and lubricant forming a very minute 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° F. On the low side, these materials have a disadvantage of increasing friction coefficient with decreasing temperature.

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° F in an air or oxidizing atmosphere, but can be used at temperatures in excess of 1200° 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 is encountered.

RADIATION EFFECTS

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

From the available literature, it appears that solid lubricants are much more radiation-resistant than the oils and grease lubricants (refs. 10-13). Clauss states (ref. 14) that the lubricity of molybdenum disulfide is not damaged by radiation, and that Teflon is damaged to a greater extent than any other lubricating plastic. He further states that Teflon is much more radiation-resistant when used in a vacuum.

Lavik (ref. 13) conducted wear life tests on five commercial solid lubricants after subjecting them to radiation levels up to 10^9 roentgens (gamma) and 3×10^{16} NVT (neutron), and found little to indicate any permanent effect on the lubricating properties of the films. The materials were of the resin- and ceramic-bonded types.

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

McDaniel (ref. 15) conducted wear life studies at 80°, 600° and 1200° F, using a Hohman A-6 (described in Chapter 6) wear tester on five high-temperature solid lubricants after they had been subjected to a

radiation dosage of 2.2×10^{11} ergs/g c and associated neutrons of 5.2×10^{16} n/cm² ($E > 2.9$ mev). He reports that reactor radiation had no significant effect on the wear life of lubricant films composed of PbS + MoS₂ + B₂O₃ or CaF₂ + ceramic binder. The wear life of a film comprised of MoS₂ + 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 Corporation (refs. 16, 17).

Hopkins (ref. 8) has tested lubricant MLF-5 (described in Chapter 5) in R-2 size bearing subjected to a radiation dosage of 10^{10} ergs/g c of gamma and 2×10^{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. 12) has reported radiation data on several plastics, including thermosetting nylon resins, polytetrafluoroethylene, and polyethylene.

LOAD AND OTHER EFFECTS

Several other effects that are not solely part of the surrounding environment must be considered when using a solid lubricant. These may include load, speed, effect of mechanical design, and operation of equipment being lubricated.

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. 18) and Hopkins (ref. 8). In general, application at high loads must be under slow-speed operation. In designing for solid lubricant film, 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 in conjunction with a binder material, exhibit decreasing friction with load, as demonstrated by Hopkins (ref. 8), Demorest (ref. 18), and Karpe (ref. 19).

Compared to other solid lubricants, the lubricating plastics generally perform best under lightly loaded conditions. 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 extremely 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.

Until recently, bonded solid lubricants were not considered suitable for use in antifriction bearings, but some work is now being done using the silicate bonded films that are showing 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.

CHAPTER 8

Novel Application Methods

There are a number of novel methods of applying solid film lubricants to component parts that have not yet been fully exploited. Quite possibly, they will become very useful in the future.

IN-SITU PROCESS

Solid lubricant film can be formed on metal surfaces from the surrounding atmosphere through the use of reactive or film forming gases. Vest (ref. 1) has discussed a method of depositing an MoS_2 lubricating film by first electrodepositing a coating of MoO_3 , then subjecting the part to H_2S gas at an elevated temperature and pressure. Film thickness can be controlled accurately by this process. For instance, a coating thickness of from 50 to 250 $\mu\text{in.}$ can be successfully applied to within $\pm 50 \mu\text{in.}$ Vest has successfully applied this coating to stainless steel 303, 304, 316, 416, and 440C and aluminum alloys of 2024, 6061 and 7075, and to both mild steel and M-10 tool steel. Vest further reports that the coefficient of friction of the film is 0.025 to 0.05, and that film wear is much better than the inorganic bonded molybdenum sulfide films. He has also applied the *in-situ* films to instrument bearing races and retainers and to instrument size gears.

ELECTROPHORETIC DEPOSITION

Coating by electrophoresis, whereby small suspended particles are deposited on an electrode with the aid of an electrostatic field, is a well-known process. It finds its widest use in the study of biological processes, where it is used as an accurate analytical device. The process, when used as discussed here, has been used for many years by the radio tube industry to coat delicate tube parts and by the rubber industry to make rubber gloves.

A solid lubricant, usually MoS_2 , can be applied by electrophoresis. The lubricant can be applied by itself or in conjunction 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, alcohol, and so forth. The lubricating solid is placed in the solvent and agitated. The formation of a good suspension is imperative, or the process will not work. There are a number of ways that the quality of the suspension can be improved, among which are (a) by the use of a finely divided lubricating material, $0.5\ \mu$ or less, and (b) by the addition of a protein material 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.

This process has been employed by a number of investigators. (See refs. 2-7.) The Vitro Laboratories have done considerable research in this field and have obtained a number of U. S. patents (refs. 8, 9) on the process. In general, they co-deposit MoS_2 with a metal oxide. The metal oxide is then reduced in a hydrogen furnace with the pure metal then being the binder for the MoS_2 coating.

Coatings applied by electrophoresis are uniform in thickness and can be deposited in hard-to-get-at areas. Apparently, very little evaluation work has been done on coatings applied in this manner, making it difficult to judge the merits of the process. However, it has been reported that the application of an electrophoretic coating of MoS_2 and boric oxide as an overcoat to an already cured ceramic-bonded solid lubricant gives wear lives many times that of the base film. Additional work is needed on this application method which appears to have several advantages.

FLAME-SPRAY PROCESS

Although the techniques of plasma and flame spraying are not new, their use as a tool for applying solid lubricant has not been fully explored. In the area of lubrication coatings, these processes have been used mainly for applying hard, wear-resistant surfaces to metal substrates. Coatings of this nature are then used in conjunction with both solid or liquid lubricants to reduce wear. Recently there has been considerable interest in the use of these techniques to apply lower temperature lubricant materials such as molybdenum disulfide.

Probably the most important feature of the flame-spraying process is the ability to apply coatings such 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 affect its

mechanical properties. As stated above, the flame-spraying process eliminates this problem.

Two processes appear useful in the applications of solid lubricants by flame spray or plasma stream techniques: (a) application of a porous ceramic coating followed by burnishing solid lubricant onto the surface and into the porous areas, and (b) application of a mixture of the binder and lubricant powder at the same time. The latter process eliminates the burnishing step, but it has some drawbacks, particularly that some of the lubricant powder is always altered by the heat required for spraying, which results in a film that is over-rich in binder, and probably in a higher friction value. Binder materials of both the ceramic or pure metal types can be used in this process.

Although there is some work being done on the application of the lower-temperature solid lubricants by the flame spray technique, sufficient data are not yet available from which to draw conclusions.

CHAPTER 9

Current Applications for Solid Lubricants

An extensive tour of government installations (NASA and military), aerospace and other industries, as well as a comprehensive literature survey, indicates that the use of solid lubricants is climbing in a great many areas. General acceptance of solid lubricants by industry, however, has been rather slow. Electrofilm, Inc., a supplier of solid film lubricants, has pointed out that its business was at one time 99 percent reliant on the military and aerospace industry. Within the last seven years, this situation has so changed that only 60 percent of its business is now with the military and aerospace industry, the rest being in the commercial field.

AEROSPACE INDUSTRY AND MILITARY

All types of solid lubricants are used in the aircraft industry on both military and commercial aircraft, as well as missiles and space vehicles. Much of this interest has been stimulated by the extremes of environmental conditions involved.

Solid lubricants are being used in over 1000 applications on the North American XB-70 aircraft. Approximately 95 percent of the B-70's plain bearings are either of the lubricating plastic or thin film ceramic-bonded solid lubricant varieties. North American is using Fabroid* in applications up to 550° F. Duroid** bearings are also employed on the B-70, but are limited to areas where the temperature does not exceed 450° F. North American is also making extensive use of its own developed ceramic-bonded lubricant material "Vitrolube," which has been shown to be a good lubricant from room temperatures up to 700° F. Applications of this material are generally on bearings of the plain bushing or plain spherical type.

* Fabroid—Teflon fiberglass material, Micromatic Hone Corporation.

** Duroid—proprietary MoS₂, Teflon-fiberglass material available from Rogers Bearing Corporation.

Solid lubricants of all types are being used extensively as the secondary lubricant on many applications on the General Dynamics F-111 aircraft. They are also using Dynalube* in conjunction with a grease containing 50 percent molybdenum disulfide on the F-111 highly loaded wing pivot bearings. Solid lubricants are also used in hinge pin applications.

Reports exist of applications of solid lubricants in missiles, satellites and spacecraft. A large number of these are of the plastic bearing variety. Goetzel et al. (ref. 1) report that a wide variety of materials has been evaluated at Lockheed Missiles and Space Company for use in space applications.

Vest and Evans at the NASA Goddard Space Flight Center, indicated that all of their work in the lubrication of satellites is directed toward the use of solid lubricants. They are primarily interested in lubricating small ball bearings and have evaluated Duroid, Bartemp, Rulon A, and a bronze Teflon material (Saylox-M).**

Evans et al. (ref. 2), Johnson and Moberly (ref. 3), Clauss and Kingery (ref. 4), and a NASA Tech Brief (ref. 5) all report applications where solid lubricants have been incorporated into electrical brushes and sliding electrical contact.

Personnel at Atomics International report more than 50 applications of solid film lubricants on gear assemblies, pins and screws, and control drum reflector assembly bearings in the SNAP 8 and 10 nuclear space reactors. In these applications, solid lubricants of the non-ceramic sodium silicate type materials are being used. Additional work reported by Kellogg (ref. 6) is available.

The United States Navy uses large quantities of solid lubricants for aircraft and shipboard applications. M. J. Devine, of the Naval Air Materiel, states that the Navy is one of the prime users of molybdenum disulfide containing greases and that their consumption of one of these materials has exceeded 100 000 lb/year.

The consensus of the military and aerospace industries contacted is that the use of these materials will increase even more in the future.

COMMERCIAL APPLICATIONS

There is also a very large market in the commercial field, including such industries as automotive, heavy electrical, metal working, and marine. The list could go on and on. It is not the intent of this document to include every application, but rather to alert the reader to the possibility of using solid lubricants in his own work.

* Dynalube—proprietary material, General Dynamics Corporation.

** Saylox-M—Allegheny Plastics.

AUTOMOTIVE INDUSTRY

According to McCabe (ref. 7), Rolls Royce and Chrysler were the only automobile manufacturers using molybdenum disulfide-containing greases prior to 1961. He further states that all Rolls Royce models are factory-lubricated with greases containing MoS_2 . Applications on the Rolls Royce include leaf springs, brake expanders, servo motors, door mechanisms, and glass winding gears.

American automobile manufacturers have also applied the use of MoS_2 containing greases for chassis lubrication. Chrysler uses concentrations of MoS_2 ranging from 3 to 60 percent in such applications as electric windows, windshield wiper assemblies, electric seat adjustments, propeller shaft splines, turn signals, and remote outside mirror applications.

The trucking and transportation industries have gone heavily to the use of molybdenum disulfide-containing lubricants. Applications include wheel bearings, king pins, chassis, and so forth. The use of molyb-

TABLE 12.—*Solid Lubricant Applications*

Applications	Lubricants
1. Radar and navigation equipment	Lubri-Bond A (Electrofilm Corporation) Metal Finishes Lubricant (Metal Finishes Company) Lube-Lok 4306 (Electrofilm Corporation)
2. Switch gear equipment	Graphite, Glycol Mixture (E. F. Houghton Company) Aqua-Dag 217 (Acheson Colloids) Molykote G (Alpha Molykote Corporation) C G-10 Graphited Alcohol Aerosol Spray (Allen Lubricants) Molykote M-88 (Alpha Molykote Corporation)
3. High-temperature oven bearings	Graphite and Glycol (E. F. Houghton Company)
4. Screw threads in manufacture of light bulbs	Aqua-Dag (Acheson Colloids)
5. Anti-seize thread applications	Aqua-Dag 217 (Acheson Colloids) Fel Pro C-5 A (Fel Pro Company)
6. Electrical appliances	Poxylube-75 (Poly-Chem Inc.) Molykote-G (Alpha Molykote Corporation)

TABLE 12.—(Concluded)

Applications	Lubricants
7. Manufacturers of electric motors	Whyte Coat 505 (Alpha Molykote Corporation) Crane Thread Lube (Crane Company)
8. Electrical equipment controls	Rulon Spray (DuPont Company) Crane Thread Lube (Crane Company)
9. Aircraft splines	Slidak (Westinghouse)
10. Installation of large generators and starters	Slidak (Westinghouse)
11. Refrigerators	Sohio STI 380 (Sohio-Standard Oil of Ohio)
12. Sliding, rolling, and oscillating surfaces in electrical motors	Lube Lok 4306 (Electrofilm Corporation)
13. Manufacturers of lamp bases and winding of electrical coils	10% Dispersion of Graphite in Glycol (Dixon Graphite)
14. Bearings for liquid oxygen applications, springs, hinges, die cast machines, and conveyors	Graphite Spray Powder #204 (Spray-on Product Inc.)

denum disulfide in a 15 percent dispersion in a solvent carrier has eliminated the problem of frozen sliders on sliding tandem trailers. There are also many other applications involving paste materials, bonded solid lubricants, and lubricating plastics.

Other automotive applications include small gears, brake and speedometer cable linings, bushings, washers, starters, generators, and alternators. The addition of MoS_2 to electric motor brushes has been investigated and adds to greatly increased brush life.

HEAVY ELECTRICAL

The heavy electrical industry is also using many solid lubricants in their products. P. H. Bowen of Westinghouse reports that they use solid lubricants and lubricants containing solids of all types throughout the organization. Some of the applications are listed in table 12.

Westinghouse researchers also have developed materials of their own. They have developed a series of lubricant composites with metal and/or lubricant matrices identified as AP, CP, HP, and G1W series. These composites have potential use in commercial applications, such as dental equipment, fluid pumps, and auto electrical systems. They are also used in the aerospace industry to lubricate bearings and gears in aircraft, space vehicles, and ground facility equipment.

METAL WORKING INDUSTRY

Bonded solid lubricants have found wide acceptance in various phases of the metal industry, as have liquid and paste materials containing solid lubricative powders, MoS_2 , graphite, and so forth.

Wide use has been made of solid lubricants for die lubrication. Liquids are often used as the carrier for ease of application, but the solids provide the lubrication. Solid lubricants also have afforded solutions to problems, such as the forming of titanium and high-strength alloys, and wire drawing.

Magie (ref. 8) has summarized the use of molybdenum disulfide in the wire industry and states that over 70 percent of the mills in this country use MoS_2 . Alpha Molykote (ref. 9) lists many industrial areas where problems have been solved by the use of solid lubricants.

CONCLUSIONS

Although from one-third to one-half of all the energy produced in the world is still lost to friction, we can expect these figures to shrink in the face of continued research on solid and other lubricants. Even a minor decrease in the friction between moving parts can, on a world-wide basis, bring about a dramatic reduction in the total energy lost to friction.

Although solid lubricants will probably never replace the home oil can, in consumer appliances solid lubricants will greatly extend the useful life of many devices the homeowner doesn't 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 further extend their 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, the solid lubricants will not displace the more conventional lubricants so much as they will open up new frontiers to operating machinery. The proper application of solid lubricants will permit the successful operation of machinery under conditions of very high temperature, cryogenic conditions, very high vacuum, nuclear radiation, extreme loads, and chemically reactive environments.

APPENDIX

The Development of Vitrolube

To show how much time and effort is required to develop a usable bonded solid lubricant, we have obtained permission from North American Aviation to include, as a part of this book, a paper presented by Mr. F. J. Williams of NAA entitled, "The Development and Testing of a New Ceramic-Bonded Dry-Film Lubricant." Mr. Williams presents the necessary steps involved and gives test results that can be used as design data.

DEVELOPMENT OF VITROLUBE

The lack of a suitable, commercial dry-film coating, and the fact that no appreciable improvement could be foreseen based on the amount of effort that was economically feasible for the dry-film vendors, have made an internal dry-film development program necessary. A few general assumptions were made at the outset of the program which limited the areas of investigation to those considered to offer the best chance of success. In general, these assumptions were:

1. Conventional pigments such as MoS_2 , graphite, silver, indium, and so forth, were satisfactory over the desired temperature range (-65° to 650° F), and no specific attempt would be made to uncover or develop new pigments.
2. MoS_2 and graphite would be incorporated as the principal lubricating pigments in all coating formulations and would be maintained at a ratio of 7 parts MoS_2 to 2 parts graphite. This ratio was decided upon because it represented the ratio most commonly used in standard dry-film coatings on the commercial market.
3. Prominently used binders would not be investigated further, thereby eliminating such materials as the epoxides, phenolics, silicates, and so forth.
4. Material and application costs would not be a limiting factor during the initial investigations.

Using these general ground rules, a concentrated program was undertaken which, within three months, led to the development of a coating

system (trade-named Vitrolube) which met or exceeded all established goals. Without elaborating on the many coatings which were formulated and found inadequate, considerable effort was spent on coatings utilizing metallic bonds, phosphate bonds, and various other types of ceramic or glass frit bonds. Some of these coatings exhibited marked improvement over the best of the commercial coatings, but did not meet the desired goal. This goal, which was established at the outset of the internal effort, was a cycle life of 20 000 cycles under the test conditions specified in the Method of Evaluation section at the end of this Appendix.

Unique features of the coating composition which can be stated in general terms are:

1. All components in the coating, including the glass binder, possess lubricating properties.
2. The ratio of binder to pigments is vastly different from what has always been considered optimum for dry-film coatings.
3. The glass binder is a combination of glasses which have not previously been utilized in dry-film coatings.

As originally developed, the processing and application techniques for the Vitrolube coating were elaborate and time-consuming. Subsequent minor changes in composition and application methods resulted in a coating that in processing and application is quite similar to standard dry-film practice. A comparison of processing procedures originally used and those presently in practice is shown in table A-1. Although, as has been stated, the present processing is similar to standard dry-film practice, the differences that exist are significant and contribute to the success of this coating. The more important of these processing features are:

PRETREATMENT

The only pretreatment utilized with this coating, other than conventional degreasing and solvent cleaning, is a precisely controlled dry-grit blast. This pretreatment was found to give equal or superior dry-film performance to all others investigated. One of the former major drawbacks to obtaining consistent dry-film performance has been a variation in the quality of other types of pretreatments (such as phosphate). By eliminating this variable, a much greater degree of reliability has been obtained with the Vitrolube coating.

DIPPING

Application of dry-film coatings by dipping procedures has been common practice within the dry-film industry for many years, but only for reasons of economy where a large number of small parts are coated at the same time. It has never been considered a proper method to obtain an optimum coating. In the case of the Vitrolube coating, the material

TABLE A-1.—*Comparison of Original and Final Processing Techniques for Vitrolube*

- | | |
|--|--|
| 1. Solvent clean. | 1. Clean by vapor degreasing or solvent wipe. |
| 2. Grit blast with 120 mesh aluminum oxide and solvent wipe. | 2. Dry grit blast with 120 mesh aluminum oxide to obtain a uniform matte finish. |
| 3. Brush on coating to a thickness sufficient to provide a finished 0.0004 to 0.0006 in. coating thickness. | 3. Solvent wipe with toluene and rinse in denatured alcohol. |
| 4. Air-dry for a minimum of 15 min. | 4. Apply coating by dipping to obtain a thickness of 0.0004–0.0006 in. The correct coating thickness is obtained by proper adjustment of the specific gravity of the coating material and rate of pullout. |
| 5. Fire in furnace for the length of time necessary for the surface of the part coated to reach required fusing temperature. | 5. Air-dry for a minimum of 15 min. |
| 6. Cool in air to room temperature. | 6. Pre-cure at 200° F for a minimum of 15 min. |
| 7. Brush on a thin layer of overcoat material consisting of glycerin, MoS ₂ , and graphite. | 7. Cure in a furnace for length of time required for the coated surface to reach required fusing temperature. Remove and allow to air-cool. |
| 8. Air-dry for minimum of 5 min. | 8. Rub coated surface with paper towel to remove loosely adhering particles. |
| 9. Fire in furnace for length of time necessary for parts to reach 600° F. | 9. Dip (or brush) a thin coat of a slurry of MoS ₂ graphite and alcohol. |
| 10. Cool in air to room temperature. | |
| 11. Burnish wear areas by rotating against mating part. | |
| 12. Brush on thin layer of topcoat material consisting of alcohol, MoS ₂ , and graphite. | |
| 13. Air-dry for minimum of 15 min. | |

was formulated specifically for application by dipping and the procedure developed to the point that optimum performance was obtained. It has been found that coating thickness can be accurately controlled by adjusting the specific gravity of the material, by varying the speed at which the part is removed from the coating material, or by a combination of both. By visual observation, dipping appears to produce a denser and smoother coating and, provided proper techniques are adhered to, thickness is uniform over the entire part and can easily be held to a tolerance of ± 0.0001 in. (nominal thickness of this coating is 0.0005 in.).

FIRING

The normal method of curing dry-film coatings is to place them in an oven controlled to $\pm 25^\circ$ F for a minimum number of hours. In the case of the Vitrolube coating, it was found to be very important to bring that part of the component being coated to a specific temperature, and that the rate at which this surface temperature was obtained was also critical. To guarantee this type of precise firing procedure and to limit the total time necessary to reach this temperature, it was necessary to

use rapid heat recovery furnaces. Thermocouples were used to accurately monitor the temperature of the coated part.

TOPCOAT

The final processing step in the application of the Vitrolube coating is the brushing or dipping of a thin slurry of MoS_2 , graphite and alcohol, which is then allowed to air dry. This topcoat can be rubbed off easily and is not of measurable thickness. Its purpose is to act as a wear-in lubricant and also to aid in the installation of close tolerance parts. Performance is definitely improved by the use of this topcoat, even though acceptable life can be obtained without its use. It is probable that the majority of dry-film coatings, and especially those which incorporate a ceramic type binder system, would show improved performance by the use of such a wear-in lubricant.

VITROLUBE TEST RESULTS

Approximately 700 separate tests have been conducted on Vitrolube-coated shaft and bushing test specimens. Many specific areas were investigated during these 700 tests, but the scope of this paper is not sufficient to discuss each of the areas studied. However, a number of the more significant areas of investigation will be discussed, along with a tabulation of test results.

BATCH CONSISTENCY

Figure A-1 is a bar chart showing the cycle life obtained with 100 separate tests on Vitrolube under conditions identical to those used during the evaluation of vendor coatings, as discussed previously. The average life for these 100 tests was 32 000 cycles, with a maximum life of 69 000 cycles and a minimum life of 14 000 cycles. The results shown are a compilation of tests conducted on each batch of material manufactured for production usage. A requirement of the NAA Process Specification for the Vitrolube coating is that a minimum of two tests be conducted on each batch of material formulated and that cycle life be no less than 20 000 cycles. A total of 23 batches of material were included in these tests. Only eight of the 100 tests produced less than the required 20 000 cycles of life, and in each of these cases new personnel were being trained who had not adequately mastered the processing techniques.

EFFECT OF SUBSTRATE

Other researchers have shown that life of dry-film lubricants is, to some extent, a function of substrate material, due either to relative hardness of the substrate or to adherence characteristics of the coating to the substrate. Table A-2 shows the results obtained with the Vitrolube coating when applied to bushings made up of 10 different substrates.

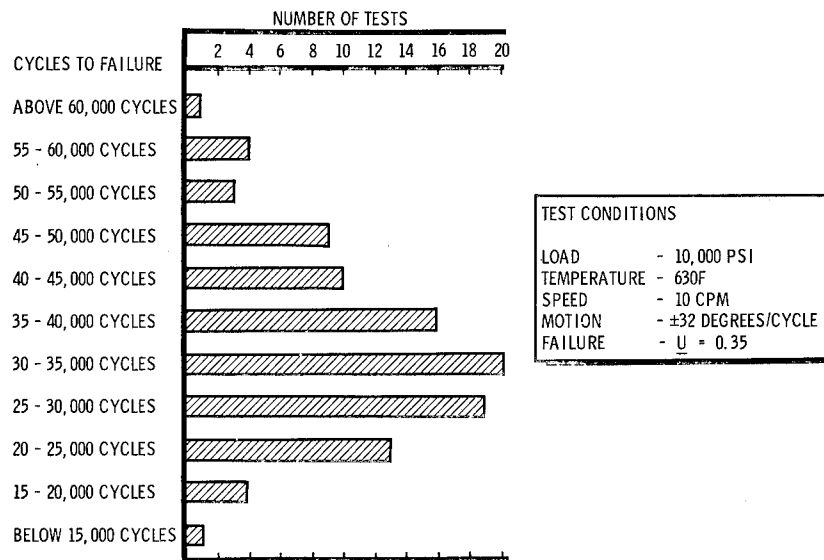


FIGURE A-1.—Summary of tests for verification of batch consistency.

TABLE A-2.—Comparison of Cycle Life with Various Substrates

Base Material of Bushing	Description of Material	Hardness, Rc	Cycles to Failure
Haynes Stellite 25	Cobalt-base alloy	36	37 869 (1)*
17-4 PH steel	Heat-treatable stainless steel	40	39 900 (2)
Beryllium copper	Heat-treatable copper-base alloy	42	17 416 (1)
Nitrided nitralloy	Low-carbon alloy steel, 0.005 to 0.010 in. nitride cases	70	32 354 (1)
S-Inconel	High-nickel alloy	28	27 064 (1)
Chromium plate (0.002 in. thick)		68	34 400 (2)
Chromium plate (0.005 in. thick)		70	26 916 (1)
Nickel-zinc alloy plate	Modified tool steel	45	25 000 (3)
H-11	Modified tool steel	54	31 000 (10)
M-2	Tool steel	64	32 000 (100)

Load: 10 000 psi; Temperature: 630° F; Speed: 10 cpm; Motion: ± 32°/cycle.

* Denotes number of tests.

In all cases, the shaft material was unchanged (chrome-plated steel). The results show that with one exception, no significant variation in life was obtained, even though the substrates varied greatly, both in terms of hardness and composition. The one exception was in the case of beryllium copper, which gave a life of less than 20 000 cycles. This poor life may possibly be attributed to an extreme loss of hardness that probably occurred during the firing process and during actual testing. Although not measured after completion of the test, it is estimated that the hardness decreased to approximately Rc20. One explanation for the consistency in results is that the same type of pretreatment was used for all substrates, thereby eliminating a variable that may have influenced the results obtained by other researchers.

EFFECT OF COATED SURFACE(S)

Based on previous test experience with resin-bonded coating, it is common practice to coat both mating surfaces to obtain optimum life. This previous experience has shown that life is increased by approximately 33 percent where both surfaces are coated, as compared to coating only the shaft (or only the bushing). The practice of coating both surfaces was, therefore, continued during the early evaluation of all vendor coatings and the internally developed coatings. To determine if coating both surfaces was beneficial in the case of the Vitrolube coating, a series of tests was conducted in which only the shaft was coated, and an additional series of tests in which only the bushing was coated.

Table A-3 is a compilation of the results obtained with the Vitrolube coating, along with results on three of the vendor coatings where similar test data have been compiled. These results are very interesting, showing that the life of the Vitrolube coating is increased by a factor of $2\frac{1}{2}$, where both surfaces are coated, as compared to coating only one surface. This significant improvement in life appears to be a characteristic of the Vitrolube coating, rather than the test conditions, since the three vendor coatings were improved by only 40–60 percent.

TABLE A-3.—*Comparison of Cycle Life—Both Surfaces Coated vs. One Surface Coated*

	Shaft Coated Only	Bushing Coated Only	Shaft and Bushing Coated
Vitrolube	13 200 (13)*	12 750 (20)*	32 000 (100)*
Code A coating (sodium silicate)	4 000 (3)	—	5 800 (30)
Code B coating	2 610 (2)	—	3 900 (12)
Code C coating	4 960 (2)	—	8000 (26)

Load: 10 000 psi; Temperature: 630° F; Speed: 10 CPM; Motion: $\pm 32^\circ$ /cycle.

* Denotes number of tests.

EFFECT OF COATING THICKNESS

Other researchers have shown that optimum life is obtained with a coating thickness of approximately 0.0003 in., with conventional resin-bonded coatings when tested at room temperature. Since a nominal thickness of 0.0005 in. had been established for the Vitrolube coating, a limited number of tests were performed on this coating in which the thickness was varied from 0.0002–0.001 in. The results indicated that as thickness was increased, cycle life was increased. This increase was quite pronounced, with almost a threefold increase in life as the thickness was increased from 0.0002–0.001 in. It is possible that this improvement in life with increased thickness is characteristic of a dry-film coating when tested at elevated temperatures. Additional tests will be required to establish the true significance of coating thickness, either in terms of the Vitrolube coating or in connection with the variation due to test temperature.

COEFFICIENT OF FRICTION

Ceramic-bonded dry-film coatings are normally considered to exhibit higher coefficient of friction values than resin-bonded coatings, especially at room temperature. Initial life testing of the Vitrolube coating showed friction values no greater and, in most cases, less than those obtained with a good resin-bonded coating. However, tests had not been conducted to specifically establish friction values for the coating. Figure A-2 shows a series of curves of coefficient of friction versus unit load at temperatures from -65° to $+700^{\circ}$ F for the Vitrolube coating. These curves show the typical dry-film pattern of decreasing coefficient of friction with increasing load. This pattern was true for all temperature levels. It is also shown that friction was highest at room temperature and was a minimum at 550° F. The curves at -30° to -65° F indicate that as the temperature decreases below room temperature, the coefficient of friction also decreases. Other types of lubricants (grease, TFE, etc.) have increased friction values at subzero temperatures. No explanation can be offered for this unusual friction trend, but similar results have been obtained by other NAA Divisions with resin-bonded dry-film coatings.

The friction values shown in this curve for the Vitrolube coating are lower than any other high-temperature coating that we have evaluated and are in no case greater than any resin-bonded coating. It should be recognized that these friction values are for the type of test specimens used in this program, and one should not attempt to compare these values with those obtained from "ring-and-block" or "pin-and-groove" type test specimens.

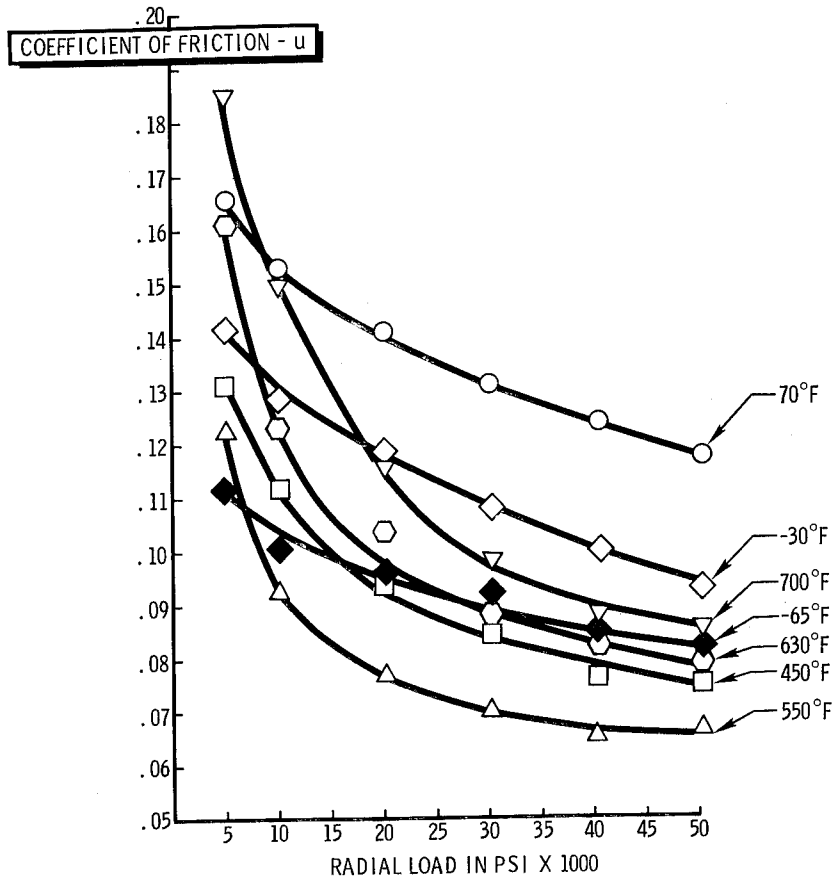


FIGURE A-2. Curves of coefficient of friction vs. load at temperatures from -65° to 700° F.

EFFECT OF TEMPERATURE

The development of the Vitrolube coating was aimed at a specific upper temperature limit of 650° F, and the coating ingredients were selected on the basis of this parameter. Further, performance at room temperature was not a prime consideration in the selection of coating ingredients. Typical air vehicle operation requires that components lubricated with a dry-film coating operate satisfactorily over the entire temperature range to which the air vehicle is subjected. To determine the performance characteristics of the Vitrolube coating throughout this temperature range, a series of tests was performed at temperatures from $+70^{\circ}$ to $+690^{\circ}$ F at a load level of 10 000 psi. The results are shown

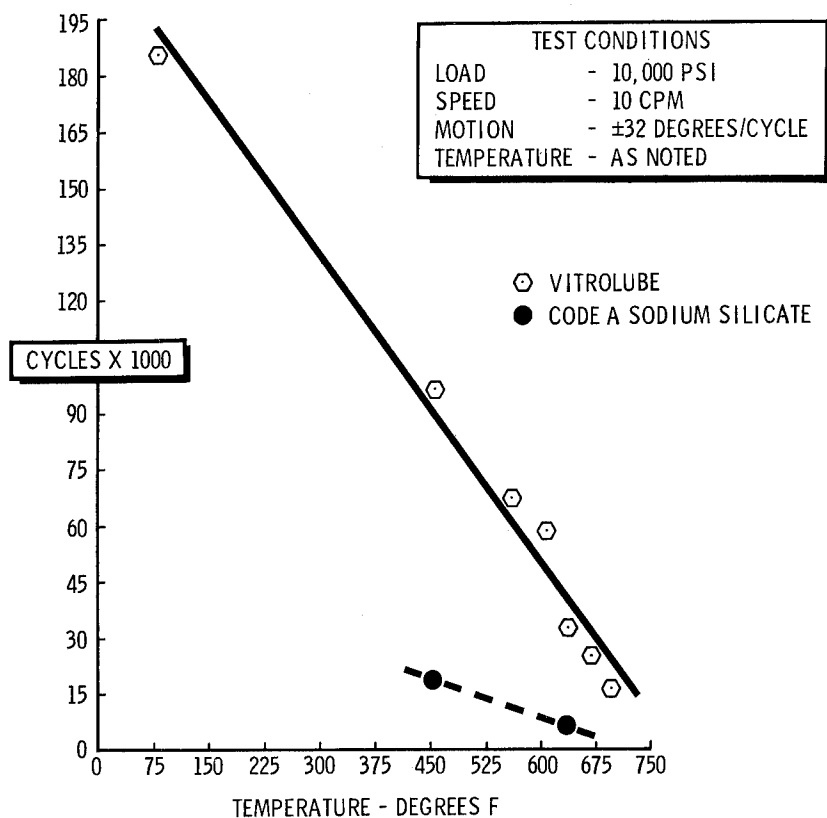


FIGURE A-3.—Curves of cycle life vs. temperature from 70°-690° F.

in figure A-3. Significant points on this curve include the extremely long life obtained at room temperature, a characteristic not typical of ceramic-bonded coatings, and the relatively long life even at 690° F, which is well above the upper limit for which this coating was developed. Also shown in this figure is the comparative life of a sodium silicate bonded coating at those temperature levels at which tests had been conducted. The Vitrolube coating is shown to have a cycle life approximately five times greater than the sodium silicate coating, where comparative data are available.

EFFECT OF LOAD

It is a commonly accepted rule that life of dry-film coatings or of any other self-lubricating surface is affected by unit loading, with life decreasing with increasing load. The slope of a curve of load versus

life will be different for different materials, and may often show a drastic change at certain load levels due to the load-carrying capacity of the material being tested. A typical life versus load curve for a resin-bonded coating at room temperature is shown in the insert on figure A-4. This curve was obtained from vendor literature in which a typical ring-and-block type tester was used. It is shown that as the unit load is decreased, life increases at a rapid rate and, further, that no drastic change in the slope occurs at any load level. To determine the effect of unit load on the Vitrolube coating, a series of tests was conducted at load levels from 2 500 to 50 000 psi and at temperatures of 70°, 450°, 550°, and 630° F.

The room temperature curve is essentially a straight line, with life increasing as the load is decreased. A significant item is the exceptionally long life at the high load levels. For comparative purposes, a good resin-bonded coating of the type approved under Military Specification MIL-L-8937 will give an average life of approximately 25 000 to 30 000

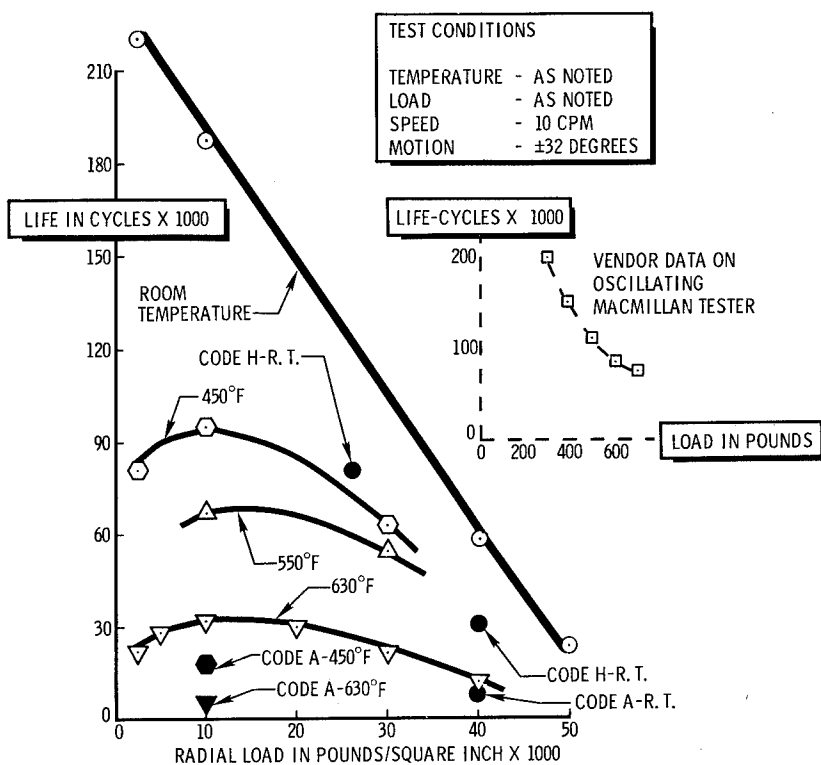


FIGURE A-4.—Curves of cycle life vs. load at various temperatures.

cycles at a load level of 40 000 psi. The Vitrolube coating gave an average life of 55 000 cycles at this load level. A typical sodium silicate bonded coating will fail in less than 10 000 cycles at this load. It can be concluded that the curve of life versus load at room temperature is in general agreement with those obtained on other coatings and with different test techniques, and agrees with the accepted load versus life rule.

The curves at elevated temperatures of 450°, 550°, and 630° F were completely unexpected and not in agreement with any previous data. These curves show that as the unit load decreases below approximately 15 000 psi, the cycle life also decreases. On the basis of these curves, life at 2500 psi is no greater than life at 20 000 psi. At unit loads above 20 000 psi, the curves follow the general pattern of decreasing life with increasing load, but with flatter slopes than the room temperature curve.

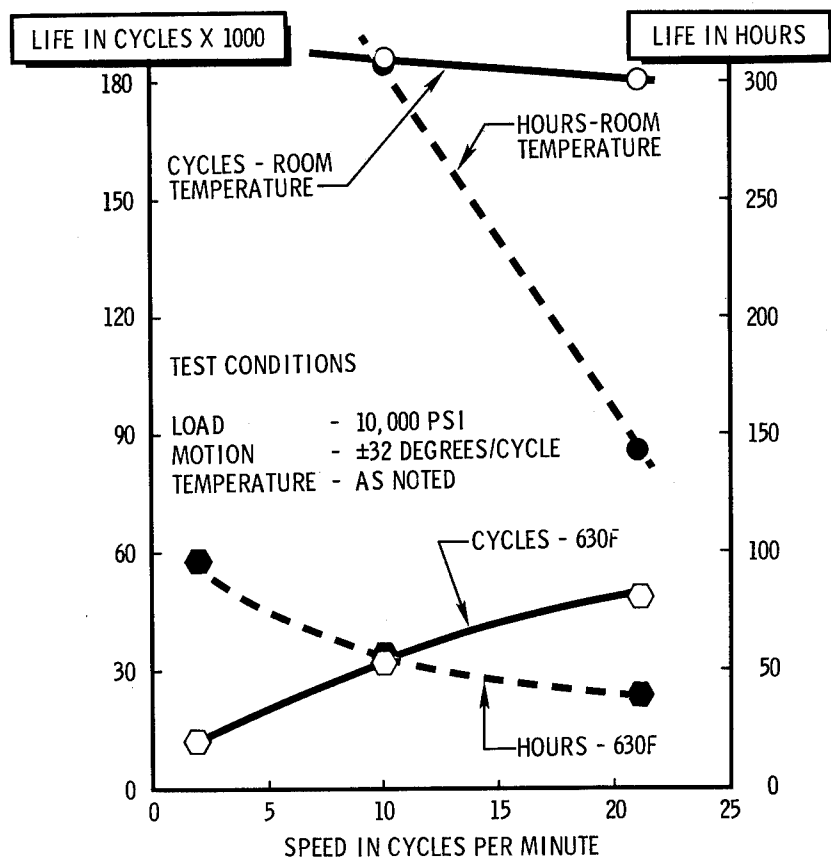


FIGURE A-5.—Curves of life vs. speed in cycles per minute at 70° and 630° F.

At the present time, no explanation can be given for this reduced life at low load levels for the elevated temperature tests. Due to the limited number of tests performed, it is possible that the normal life scatter of this coating has abnormally influenced these curves. However, sufficient data have been accumulated to show that lower unit loads will result in no appreciable increase in life. This may be a characteristic of the Vitrolube coating, or it may be a characteristic of all dry-film coatings when tested at elevated temperatures.

Also shown in figure A-4 are results obtained with some of the vendor coatings at those load levels and temperature levels at which tests had been conducted. In all cases, the Vitrolube coating is shown to have greatly superior life at all load and temperature levels at which a comparison is possible.

EFFECT OF CYCLE RATE

The 10-cycle-per-minute speed that was used as one of the basic test parameters was selected because of previous experience at this speed during earlier dry-film and grease test programs, and because this speed was representative of many air vehicle applications. To determine the effect that cycle rate would have on the life of the Vitrolube coating, a limited number of tests were performed at cycle rates from 2-21 cpm at room temperature and at 630° F. In figure A-5, the results of these tests are plotted as curves of speed versus life in cycles and speed versus life in hours.

The room temperature curves show that life in cycles remains relatively unchanged, regardless of the rate of cycling, and that, conversely, the life in hours is reduced (or increased) in a direct ratio to the rate of cycling. This indicates that at room temperature, accelerated testing is feasible in those cases where life is listed in terms of total cycles to failure. However, the 630° F temperature curves show that as the cycle rate is increased, the life in cycles also increases, and that this increase is appreciable. Specifically, at a rate of 2 cpm, the cycle life was 13 000 cycles, and at 21 cpm, the cycle life was 47 000 cycles, with all other test parameters unchanged. Life in terms of hours decreases as the cycle rate is increased, but not in a direct ratio to cycle rate as existed at room temperature.

One significant conclusion that can be drawn from these results is that accelerated testing at elevated temperatures is not feasible unless sufficient prior data are available on the coating being tested to allow for interpretation of the results obtained. The explanation for these results is that the coating, as has been shown in figure A-3, has decreased performance with increasing temperature. Therefore, both the total time at which the coating is subjected to temperature during cycling and the rate at which cycles are accumulated per unit of time while at tem-

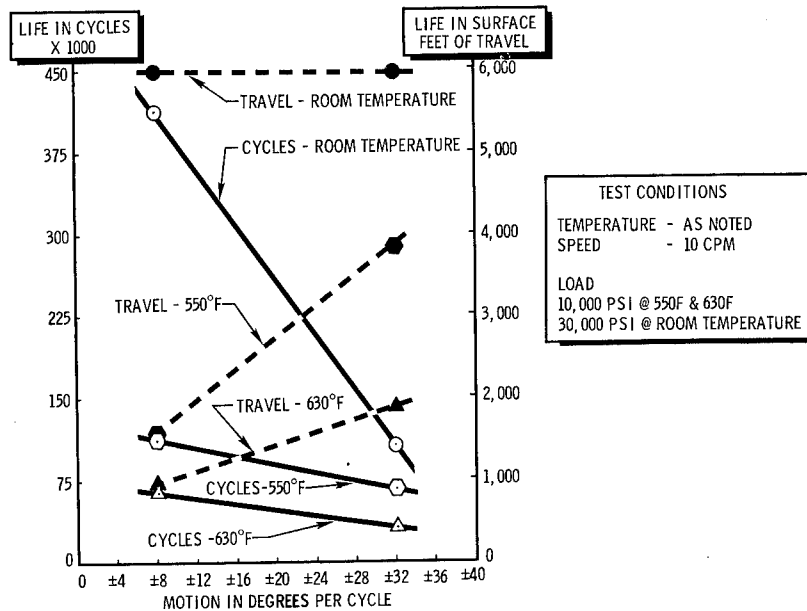


FIGURE A-6.—Curves of life vs. degree of motion at 70°, 550°, and 630° F.

perature will influence the life of the coating. By increasing the cycle rate, the coating is subjected to a fewer number of hours at temperature in accumulating a given number of cycles, and, therefore, the cycle life of the coating will be increased. More extensive testing is needed to establish a more precise life versus speed relationship at various temperatures; the above comments are made only to show that the choice of cycle rate can strongly influence results during elevated temperature testing. As an added comment, it is probable that, even at room temperature, those test procedures which impose high loads and high speeds generate sufficient frictional heating to affect test results adversely, especially on those coatings with marginal temperature resistance.

EFFECT OF MOTION

The $\pm 32^\circ$ of oscillation that was used as one of the basic test parameters was also selected because previous test programs had been conducted using this amount of motion, and because this value, combined with the 10-cpm cycle rate, resulted in a surface speed of approximately 0.6 ft/min for the size of specimens being tested. Most air vehicle components have surface speeds between 0.1 and 1.0 ft/min. To determine the effect of degree of motion on life of the Vitrolube coating, a series of tests was conducted at motions of $\pm 8^\circ$ and $\pm 32^\circ$, and at

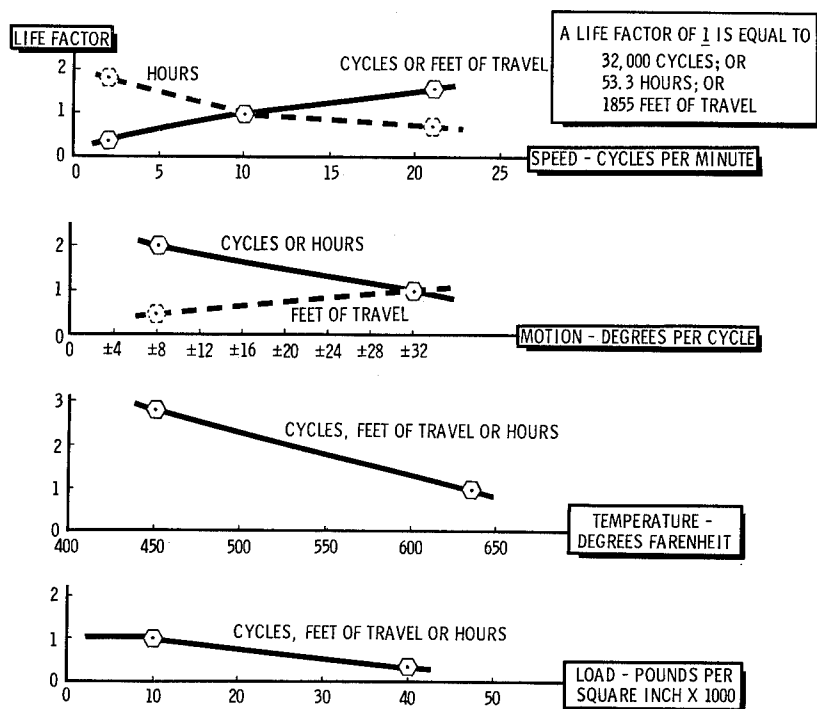


FIGURE A-7.—Life factor curves.

temperatures of 70°, 550°, and 630° F. Curves plotted from these results are shown in figure A-6, in which life is shown both in terms of cycles and of feet traveled.

At room temperature, life in terms of feet of travel is unchanged, regardless of the degree of motion, and, conversely, life in cycles increases with decreased motion in a direct ratio (that is, a 4:1 decrease in degree of oscillation results in a fourfold increase in cycle life). This indicates that total feet of travel is the best criterion to use at room temperature in those cases where different test techniques may produce varying amounts of travel per cycle. However, at elevated temperatures, the curves no longer follow this pattern, again due to the adverse effect of elevated temperature on the coating. As a direct comparison to room temperature, a 4:1 decrease in degree of oscillation at a test temperature of 630° F results in only a twofold increase in cycle life, and, at the same time, life in feet of travel is reduced by a factor of two.

SUMMARY OF TEMPERATURE, LOAD, SPEED, AND MOTION EFFECTS

In summarizing the results shown in figures 3 through 6, it is readily apparent that many factors must be considered when analyzing dry-

film test data, and especially where such data are being used to predict or guarantee the satisfactory operation of a dry-film coating in an air vehicle component encompassing a variety of operating conditions. From these tests it can be concluded that a great deal of additional test data are needed before accurate predictions of performance of the Vitrolube coating can be made for any specific application. However, this is also true for all other dry-film coatings and, in most cases, to a much greater extent. Figure A-7 is a series of life factor curves for the Vitrolube coating for the variables that were investigated—load, temperature, speed, and motion. In most cases, only a few points were available to establish these curves, and, even then, the number of tests was not sufficient to guarantee that these curves are accurate. They are presented only as one way in which dry-film test data can be used to predict life at any given set of operating conditions, provided enough test data are available to be sure that all curves are valid.

MISCELLANEOUS TEST INFORMATION

Initial evaluation of the Vitrolube coating by other organizations for possible use under extreme environmental conditions has produced promising results. Significant conclusions made on the basis of these tests were:

1. At reduced pressures of approximately 10^{-7} torr, the Vitrolube coating gave longer life and lower coefficients of friction than any dry-film coating tested.
2. At cryogenic temperatures of approximately -200° F the Vitrolube coating gave extremely long cycle life, and friction values were less than those at room temperature. No comparison can be made with other coatings, as none were evaluated under these conditions.
3. The elimination of the graphite component of the coating has no appreciable effect on the life of the Vitrolube coating. This may not be true under all test conditions, and would require verification if an all MoS_2 coating were contemplated.

CONCLUSION

The Vitrolube dry-film coating has undergone extensive testing under conditions closely representing actual air vehicle usage. Results have shown this coating to have a life greatly superior to that of all other coatings tested to 690° F with the type of evaluation techniques used. However, the Vitrolube coating has the same drawbacks that must be recognized in the use of any dry-film coating. The coating, as is true of any other dry-film coating, does not inhibit corrosion, and completely corrosion-resistant substrates must be used to eliminate this problem. Contamination of the wear surfaces by any type of fluid, and especially by the type of synthetic fluids used in supersonic air vehicles, is detri-

mental to the coating, although not to as great an extent as with more conventionally bonded coatings. Sharp edges, careless handling, and abrasive type contaminants will cause drastic reduction in wear life, and extreme care must be observed if the coating is to perform in service as well as under laboratory conditions. The coating is life-limited and cannot be replenished in place, so that replacement of the coated components must be accomplished at preset intervals. These are some of the problems that must be recognized in the design and use of components incorporating the Vitrolube coating.

By recognizing these problems and by incorporating appropriate design practices, the Vitrolube coating has given very satisfactory performance in every evaluated application.

METHOD OF EVALUATION

The testing of lubricants, specifically the testing of dry-film coatings, has always been one of compromise and in which agreement on technique or method has been lacking among users, producers, researchers, and so forth. The only positive method of evaluation which could guarantee satisfactory performance in service would be to conduct a life test on each actual part or system under operating and environmental conditions corresponding to actual air vehicle operation. Obviously, this is unacceptable where many parts are involved, due to the cost of testing and the length of time that would be required. At the other end of the scale are so-called bench type testers which are inexpensive to operate and give fast and plentiful test results. However, these testers produce data which are comparative only, and are not representative of actual air vehicle application. The various forms of ring-and-block and pin-and-groove type testers are examples of bench type testers.

The selected method of evaluation was based both on prior testing experience, which had allowed satisfactory selection of lubricants for lower temperature applications, and the availability of test equipment for the method chosen. Figure A-8 is a sketch of the type of test specimens used and the mode of operation of the test equipment. This figure shows that the test specimens consisted of a shaft and bushing similar in design and material to that which would be used in an actual vehicle application. The specific operating conditions were chosen such that they best represented conditions that would exist in service and at the same time could be accomplished without undue cost and/or test time. These test conditions were:

1. Load: 3910 lb applied radially, which, for the 5/8-in. bushing bore, resulted in a unit load of 10 000 psi over the projected bearing area.
2. Speed: 10 oscillating cycles per minute.
3. Motion: $\pm 32^\circ$ per cycle (this means that starting at the mid-

point, the shaft is rotated 32° in a clockwise direction, then back to the midpoint, then 32° counterclockwise and back to the midpoint).

4. Temperature: 630° F at the interface of the shaft and bushing.

5. Duration: Until reaching a coefficient of friction of 0.35 (this value was based on previous tests which had shown that at this friction value, slight metal-to-metal contact was occurring under these specific test conditions).

TEST SHAFT	
MATERIAL:	H-11 STEEL
HARDNESS:	ROCKWELL C 54
PLATING:	CHROME (0.002 IN. THICK)
SIZE:	0.6230 IN O. D. X 3-5/8 IN. LONG

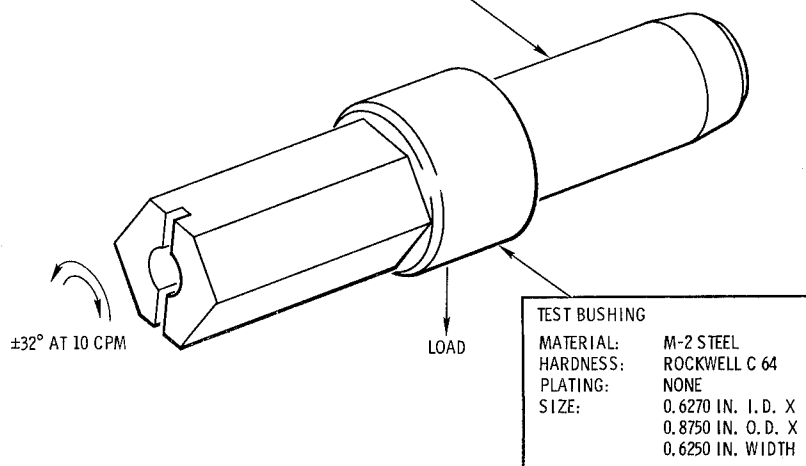


FIGURE A-8.—Test specimen configuration.

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CHAPTER 3. SPECIFICATIONS

1. MIL-L-8937 (ASG) Lubricant, Solid Film, Heat-Cured.
2. MIL-L-23398 (ASG) Lubricant, Solid Film, Air Drying.
3. MIL-L-46010 (MR) Lubricant, Solid Film: Heat-Cured, Corrosion Inhibiting.
4. Federal Test Method Standard No. 791
 - a. Method 3807—Endurance (Wear) Life of Dry Solid Film Lubricants.
 - b. Method 3810—Adhesion of Dry Solid Film Lubricants.
 - c. Method 3812—Load-Carrying Capacity, Dry Solid Film Lubricants.
 - d. Method 4001—Protection, Salt Spray (Fog).
5. MIL-L-81329 Lubricant, Solid Film, Extreme Environments (WEP).
6. Federal Test Method Standard No. 791, Method 333. Performance characteristics of Lubrication Grease in Antifriction Bearings at Elevated Temperatures.

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