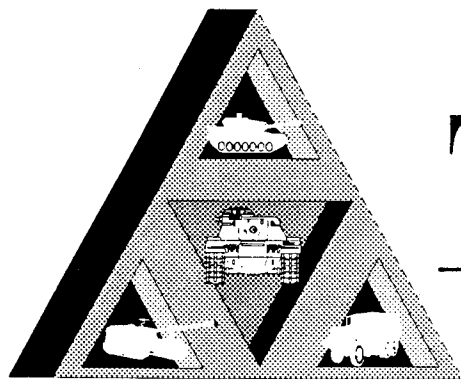
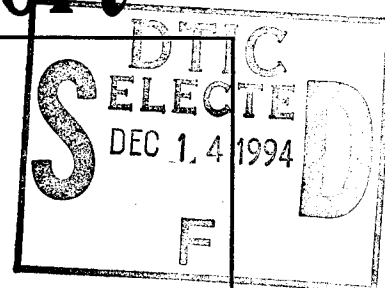


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Technical Report

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Information Compendium on Solid Film Lubricants

November 1994

By **Tonie S. Seide**

USA Tank Automotive Command
Mobility Technology Center Belvoir

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Section 1 Introduction

Solid film lubricants have become quite beneficial in lubrication technology. The use of solid film lubricants help alleviate common lubrication problems encountered with "conventional lubricants." Unlike solid film lubricants, oils and greases tend to attract dirt and other abrasive particles which can lead to increased friction and wear as well as frequent repair and maintenance. While oils and greases function well under limited conditions, solid film lubricants can perform under extreme conditions and environments when used appropriately.¹ Hence, solid lubricants can save space, weight and money by diminishing the need for maintenance and complicated lubricating systems.

A solid film lubricant is a non-liquid material which is applied to the surface of a metal substrate to provide lubrication and minimize friction and wear of moving elements. Solid lubricants are utilized in several forms such as loose powders, bonded films, composites and laminates. Each form of lubricant has distinct characteristics which lead to the diversity of uses for solid film lubrication.² Powdered solids exist today as the simplest form of solid lubricant. In powder lubrication, a thin film is formed by direct application of a loose powder to a surface needing lubrication. However, powdered lubricants are generally restricted to applications in which lubricant adhesion is not essential.^{1,3a}

Bonded films, on the other hand, consist of lubricating pigments as well as binder materials which help distinguish these lubricants from other lubricating solids. Bonded lubricants can be of an organic or inorganic nature and are designated as heat/air-cured resin bonded lubricants or ceramic/non-ceramic bonded lubricants, respectively. Air-cured resin-bonded solid lubricants employ thermoplastic resins as bonding agents. These thermoplastic binders include acrylics, cellulose, vinyls and alkyds which require no heating yet yield a fairly hard coating.^{3a} However, these resins do not offer good solvent resistance.^{3a} In contrast, heat-cured resin bonded lubricants require baking since thermoset resins such as epoxies, epoxy-phenolics, silicones, polyamides and polyimides are used as the binding materials. These heat-cured materials provide greater load capabilities and wear life than the air-cured lubricants and are more commonly used in solid film industry.^{3a}

Inorganic-bonded solid films are high temperature lubricants which contain ceramic or salt-based bonding agents. Ceramic-bonded lubricants employ glass binders instead of resins and are useful at temperatures ranging from 500°F to 1500°F and beyond with their most significant attribute being good strength at increased temperatures.^{3a} Non-ceramic lubricants utilize water soluble silicate and phosphate binders. Generally speaking, these lubricants function at temperatures from -300°F to +1000°F with minimal outgassing under hard vacuum as well as liquid oxygen compatibility.^{3a} However, non-ceramic bonded films do not provide adequate corrosion protection and become soft when exposed to water or moisture for a long duration.^{3a}

Composite lubricants have brought a new dimension to solid film technology. Composites are derived by combining lubricating pigments and other material powders such as metals and plastics to form a compact solid body. These composite materials provide effective lubrication by transference of the solid lubricant within the compact to the surface needing lubrication. These compacted lubricants can be used as bearing components, cage materials for bearings, electric brush material and for lubricating gears and similar equipment.

Laminate solid film lubricants are usually composed of layers of cloth, fiber glass or nylon fabric, in addition to resin and a lubricating solid such as molybdenum disulfide, graphite or Teflon. These laminates are compressed under pressure and heat, and exhibit high-bearing strength and hardness. However, the resin and fabric composition of these materials limit their range of operating temperatures (max. 400°F).^{3b}

Section 2 Background

Currently the U.S. Army uses solid film lubricants that are qualified in accordance with military specifications MIL-L-23398, MIL-L-46010, MIL-L-46147, and MIL-L-81329. These military specifications establish guidelines for air-cured, heat-cured, and extreme environment solid film lubricants that are procured and utilized for wear reduction, corrosion protection, and prevention of galling and seizure of metals. Solid film lubricants can be applied on substrates such as aluminum and its alloys, copper and its alloys, steel, titanium, stainless steel, and chromium and nickel bearing surfaces. The U.S. Army often uses solid lubrication on weapons, ground vehicles, and ground handling equipment. For instance, the Army uses solid film coatings in the field to touch up worn surfaces and on inaccessible parts where conventional oil and grease lubricants are difficult to apply. The Army also uses thin film lubricants for sliding motion applications and under conditions where heavy-load capacity, solvent resistance, and long-term corrosion protection are needed. Some specific Army applications include use on components of the XM34, rocket launcher; XM3E2, Loading Platform, truck-mounted; XM449, trailer; XM552, trailer; XM505, trailer; M79, 106 Rifle Mount; XM32, Rocket Launcher; and XM31, 105 MM Howitzer.⁴

Since solid film lubricants have proven to be effective for many applications, it has been proposed that new solid film lubricants be evaluated and/or formulated for future Army ground materiel lubrication needs. While the U.S. Army needs a multi-functional coating which provides lubrication and corrosion protection for ground vehicle equipment, this need is coupled with a desire for environmentally safe lubricants that can be applied during depot maintenance or at time of manufacture and require little or no maintenance. Hence, new developments should focus on high performance-low VOC (volatile organic components) solid film lubricants that meet emerging air quality standards and function under oscillatory, sliding, or rolling conditions. These solid film lubricants should be able to withstand exposure to corrosive environments such as high humidity, salt spray and chemical reaction products. Also, viable candidates should possess qualities such as a low coefficient of friction, high load-carrying capacity, good corrosion resistance, extended wear life, a wide temperature range, and be free of hazardous components such as lead (Pb) or antimony (Sb). Finally, the lubricants must be able to be applied to lubrication contact points such as road wheels, trunnion bearings, suspension joints, flap tracks, cam surfaces, plain and spherical bearings, threads, gears, gunsights, gun elevating mechanisms, and recoil mechanisms.⁵

Section 3 Formulation of Bonded Solid Film Lubricants

Although new technologies are emerging everyday, bonded solid film lubricants are the most commonly used in the solid film industry. As stated previously, the formulation of a bonded solid film lubricant incorporates a binder and lubricating pigment in a solvent system which acts as a diluent for the lubricant dispersion and facilitates application methods such as spraying, dipping, rolling, etc. The lubricating pigment's primary function is to reduce friction and wear while the binder serves as its carrier providing adhesion between the solid film coating and the metal substrate. Additives such as flow agents, corrosion inhibitors, dispersants, and color pigments may then be employed to achieve a low coefficient of friction, corrosion resistance, solvent resistance, radiation resistance, high load-carrying capacity or other desirable properties.

Many parameters influence the quality of performance of the final lubricant composition. These include the selection of a lubricating pigment, the adhesive and solvent system chosen, the pigment-to-binder ratio, and the particle size of the lubricant material. The condition of the substrate, type of motion, curing process, and environmental and other operating conditions are also very important and shall be discussed in later sections.

Initially, the need and use for a solid film must be established to assist in selection of its essential components. When selecting a lubricant material, the intended use as well as the merits, deficiencies and compatibility of the pigment and binder to be used must be considered since these characteristics make some lubricants more suitable for certain applications than others. For example, molybdenum disulfide, graphite, and polytetrafluoroethylene (PTFE) all have substantially low coefficients of friction, but each has unique capabilities. Molybdenum disulfide has a broad temperature range but is ineffective at light loads and in moist or radioactive environments. Graphite provides lubrication at high speeds in the presence of moisture but is electrically conductive and can cause corrosion. Finally, PTFE is only suitable for low speed applications at light loads and limited temperatures.

Similarly, binders, having distinct qualities of their own, are selected based on their compatibility and ability to complement the lubricant medium. For instance, non-ceramic binders such as silicates lack corrosion protection and chemical resistance, but they have high temperature capabilities and liquid oxygen(LOX) compatibility whereas organic binders like epoxies are heat-cured resins which provide good solvent resistance and corrosion protection.^{3a}

Basically, the bonding constituent is the governing factor in choosing a solvent system since knowledge of the solubility and evaporation rate of the resin is necessary for optimum dilution of the lubricant dispersion (lubricating pigment and bonding agent). Any solvent from water to methylene chloride can be utilized as a diluent. However, the solvent must be compatible with the lubricating pigment and substrate material as well as

the adhesive binder selected. Also, the solvent must have good storage stability, a sufficiently high flashpoint and comply with environmental regulations (EPA & OSHA) to qualify as a suitable candidate.

In formulating a solid film lubricant, one must achieve the proper ratio of the lubricating and bonding components as well as determine the optimum particle size of the lubricative pigment. Researchers have found that particle size affects the corrosion protection of the solid film,⁴ and that varying the percentage of lubricating pigment to bonding agent significantly affects the overall properties of the coating.⁹ A low pigment to binder content usually produces a glossy, hard, durable film with a relatively high coefficient of friction and good corrosion resistance. Conversely, a high percentage of pigment to binder content yields a dull, soft, powdery film with a low coefficient of friction and poor corrosion resistance. Since there is such a wide range of possibilities between the two extremes, the optimum ratio is often obtained by trial and error. However, the proper pigment to binder ratio depends on the nature of intended use and the type of ingredients being combined. Furthermore, insufficient binder or pigment content will result in poor bonding and lubricity, respectively.

Section 4 Substrate Characteristics

The effectiveness of a solid film coating depends on the quality of the substrate to which it is applied as well as the nature of contact between the mating surfaces. Generally, the surfaces of metals/alloys encountered throughout industry are contaminated by some form of reaction with their environment or physical contact. The subsequent formation of chemical, oil, grease or adsorbed films (containing primarily water vapor and oxygen from the atmosphere) vary from metal to metal influencing frictional properties. Likewise, other properties including the hardness and surface finish of the substrate affect the wear-life and friction coefficient.

Usually, pure metals exhibit low hardness. However, their hardness and wear resistance can be increased by alloying or heat treatment. The hardness of a substrate is commonly measured by the Rockwell, Vickers diamond pyramid, or Brinell testers under a definite load and time although other testing techniques exist which employ scratch hardness.¹⁰ Hardness is indicative of the strength, workability, and machinability of a material and is generally determined to compare similar materials under similar conditions. Hard substrates usually limit wear and surface damage while decreasing friction since hard substrates provide rigid support whereas soft materials undergo greater deformation.

Surface finish impacts the adhesion between the substrate and lubricant film. Many times the surface finish produced during manufacture must be altered by some form of substrate pretreatment to attain a desirable surface texture. Since research indicates that roughened surfaces improve adhesion and result in increased wear life,^{11,12} surface roughness is desired to a certain extent. Although rough surfaces are preferred for instance when galling or high friction temperatures are probable, smooth surfaces should be considered when smooth motion or accurate dimensions of the surface component are required.¹³

The type of contact which exists between mating surfaces also influences the wear life of the lubricant film. Point or line contact exists when mating parts touch at a single point or along a straight or curved line, respectively. Point or line contact under rolling conditions is exemplified by ball and roller bearings, respectively while crowned cams and plain bushings illustrate point and line contact under sliding conditions. The contact area is expanded when loads are induced which can increase the wear rate and reduce wear life.

Section 5 Surface Preparation

A substrate is pretreated to promote good adhesion between the metal and a solid film lubricant as well as extend lubricant wear life and corrosion protection. The degree of adhesion is dependent upon the surface pretreatment. The majority of failures of solid film lubricants are the result of inadequate surface preparation or misapplication.^{14,15} Consequently, different pretreatments are recommended for different substrates depending on the nature of the substrate and the desired lubricant performance namely increased corrosion or wear resistance.^{4,10,16} For example, phosphatized coatings significantly increase wear life and are recommended yet are best suited for iron and steel substrates.¹⁷

Generally, pretreatment involves a cleaning process such as vapor degreasing, solvent wash or bright dipping to remove surface contaminants which can impede film adhesion. Next, the cleaning is followed by surface alteration to increase the surface area. This may be accomplished via grit or vapor blasting, chemical etching, etc. Finally, the substrate is treated with a preliminary coating by means of phosphatizing, anodizing, black oxidizing or passivation in order to provide or enhance necessary protection.^{10,14}

While pretreating substrate materials offers significant advantages, certain limitations do exist. As stated previously, the type of substrate employed will determine the pretreatment technique. Lack of or poor surface pretreatment can be detrimental to lubricant performance. Additionally, since some pretreatment techniques may be limited in their method of application or require accurate control, operator variability increases inconsistency and the possibility of lubricant failure.

Section 6 Coating and Curing Processes for Solid Lubricants

Many coating processes have been developed for applying solid film lubricants to substrate materials. Since successful operation of a machine relies on proper application of the solid film lubricant, the method of application selected should be based on parameters such as equipment design, lubricant composition, quality of adhesion, desired film thickness, intended use, and cost and availability.

Resin bonded lubricants are normally applied by rolling, dipping, brushing, tumbling, or spraying which is the most common and effective method for achieving optimum performance. Conventional spray techniques allow the air pressure and solids content of the lubricant formulation to be regulated providing smoothness, uniformity, and the appropriate thickness (.0002in. - .0005in.) which is critical to lubricant wear life.¹¹ Dipping techniques are utilized when design configurations make coating by spray method difficult and inadequate. Tumbling techniques can be used for coating large quantities of small parts in order to minimize handling and cost.

Other lubricating solids may be applied by various methods which include burnishing, sputtering, vapor deposition, and plasma spraying. Application of lubricating powders by burnishing usually involves rubbing with a mechanical means of force to obtain a thin film. However, this method which relies on the lubricant and substrate's affinity for one another provides marginal adhesion and thickness is not easily controlled.^{2,18}

Sputtering is a vacuum deposition process which atomically cleans and etches the substrate material providing very thin films with excellent adhesion.² Sputtering eliminates the need for binders and does not lead to dissociation of the lubricant composition that may occur with certain lubricating solids during physical vapor deposition.^{2,19} Physical vapor deposition is a more common deposition technique but it is not suitable for alloys and lubricating pigments such as molybdenum disulfide (MoS_2).¹⁹ The Plasma-Spray method is a thermal deposition technique which enables ceramics and other high temperature solid film lubricants to be applied to metal substrates without affecting mechanical properties.²⁰ Also, the plasma-spray process eliminates the use of diluents (organic solvents/water) and the need for a curing process.

Most resin-bonded lubricants do require a curing process for bonding which may involve air-curing(thermoplastic resins) by solvent evaporation or heat-curing(thermoset resins) by crosslinking. Since the curing conditions vary according to the type of base metal and binders utilized, curing temperatures may range from room temperature to 2000°F.^{3a,4} Likewise, curing times may range from a few minutes to several hours.^{3a,4} During the curing process metals/alloys can be weakened if mechanical properties are altered by curing temperatures which exceed the maximum temperature limit of the substrate material. Similarly, certain binders deteriorate at elevated temperatures. For, example some aluminum alloy compositions cannot withstand curing temperatures beyond 275°F,^{3a} and common organic binders deteriorate at curing temperatures above 450°F.^{3c}

Curing conditions are important for complete cure and optimum lubricant performance. Higher curing temperatures may increase wear-life but lower curing temperatures provide better corrosion protection.^{4,21}

Section 7 Impact of Environmental and Operational Conditions

The environmental and operating conditions which are imposed upon the surfaces in motion affect the performance of a thin film coating. Most lubricants are restricted to use in a specific environment (air, vacuum, radioactive, etc.) because their lubricating properties are impaired by changes in atmosphere. For instance, many lubricants are incompatible with liquid oxygen. Also, lubricants that require some form of moisture for lubrication are ineffective in vacuum environments even though researchers have found that a vacuum atmosphere can increase the wear life of certain lubricants.²² Research also indicates that radioactive environments have little effect on a lubricant's wear life, corrosion resistance, or thermal stability.^{3c,4,17}

Under normal conditions, lubricants may encounter contaminants which can adversely affect wear rate, wear life, or corrosion protection. Dirt and dust particles are examples of abrasive contaminants which increase the wear rate and can induce abrasive wear. Similarly, environmental contaminants such as humidity, salt water or chemical reaction products (eg. sulfurous acid) create corrosive environments which can react with the substrate and/or lubricant materials and lead to corrosive wear. The presence of these contaminants or others such as conventional lubricants can cause damage to a lubricant film significantly decreasing wear life, corrosion resistance, and the lubricant's ability to provide effective lubrication.^{3c,3d} Conventional lubricants such as oils and greases can increase friction and structurally weaken bonded films. Although simultaneous use of conventional lubricants and bonded films is possible when compatible, research has shown that oils and greases diminish the load-carrying capacity and wear life of bonded lubricants.^{3d,9}

The temperatures, loads and speeds encountered during operation compromise the integrity of thin film coatings with temperature and load having the most pronounced effect on a lubricant's wear life.¹⁷ Usually, high temperatures reduce wear life and cause degradation of some solid film lubricants. Since load is directly related to wear - the wear rate increases with load. But speed has little or no effect on wear life unless the speed is high enough to deteriorate the binder utilized.^{16,17,23} Consequently, excessive loads and/or speeds can destroy a solid film coating and result in severe surface damage.

Section 8 Methods of Evaluating Solid Film Lubricants

Wear-life and friction testing provide a means of evaluating the load carrying capacity and endurance of solid film lubricants. Friction and wear testers are designed to simulate the contact pressure(load) and type of motion occurring between surfaces. A spring, hydraulic, or dead weight load may be applied to specimens generating point, line, or larger area contact depending upon the configuration of the tester. Surveys have been conducted to identify friction and wear testers which have been developed.^{24,25,26} Testers, such as Falex, McMillan, etc., differ in capabilities as well as configuration. Typical capabilities include operation under sliding and/or rolling conditions; in oscillatory, rotary, or unidirectional motion; and with constant or variable speeds. Measurement of the friction and wear characteristics can provide determinations for kinetic and static coefficients of friction, wear life, and wear rate. However, it is difficult to show correlation, repeatability and reproducibility of data from differing apparatus.

Other methods of evaluating solid film lubricants involve standard testing and surface examination. Standard tests have been developed to determine characteristics such as hardness, adhesion, film thickness, particle size, corrosion and chip resistance, etc. Braithwaite²⁷ gives an excellent overview of the techniques for examining surfaces. The surface texture and other surface properties can be identified with the aid of equipment such as stylus instruments, microscopes, electron diffraction, thermal analysis and others. For instance, the Revetest, a scratch test apparatus, was designed to measure the mechanical strength of hard or brittle coatings. Differential Thermal Analysis (DTA) and a Thermomechanical Analyzer (TMA) can be used to detect chemical reactions and dimensional changes, respectively.

Although a wide range of equipment and tests exists for evaluating solid film lubricants, the solid film industry lacks standardized test machines for lubricant development which makes repeatability and correlation of testing difficult. Generally, testing machines are designed to simulate service conditions. While laboratory testing may serve as a good estimate of a lubricant's potential, laboratory equipment cannot accurately control or reproduce all of the environmental and operating variables needed to accurately predict service performance.

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Appendix A Lubricating Pigments

Lubricating Pigment	Temperature (°F) Limit	Load Capacity (psi)	Strengths and Weaknesses
MOLYBDENUM DISULFIDE (MoS ₂)	-400 to 750 (Air) 500 (N ₂ /Vac)	>100,000	Low coefficient of friction with lower friction in vacuum; unsuitable for radioactive environments; ineffective at very light loads; promotes corrosion in presence of moisture; poor oxidation resistance.
POLYTETRAFLUOROETHYLENE (PTFE)	-100 to 550	10,000	Lowest coefficient of friction; chemically inert; electrically conductive; only suitable for low speed applications; used for cosmetic coatings.
FLUORINATED ETHYLENE PROPYLENE (FEP)	-100 to 400	<10,000	Low coefficient of friction; intended for light load applications.
GRAPHITE	-400 to 1200 (Air) Unst. (Vac)	40,000	Lubricates at light loads and high speeds; electrically conductive; poor corrosion resistance.
TUNGSTEN DISULFIDE (WS ₂)	-400 to 800 (Air) 1500 (N ₂ /Vac)	≈100,000	Relatively stable and inert; behaves same as MoS ₂ with a lower coefficient of friction and better oxidation resistance.

Appendix B Bonding Agents

BINDER MATERIAL	USEFUL TEMP. RANGES °F	STRENGTHS & WEAKNESS
Thermoplastic: Acrylics Cellulosics Vinyls Alkyds	-100 to 350	Good corrosion resistance and durability; poor solvent resistance; poor outgassing properties; and poor radiation resistance
Thermoset: Epoxies Phenolic Epoxy-phenolics Silicones Polyamides Polyimides	-320 to 500	Excellent corrosion and chemical resistance; excellent durability-hardness, chip and abrasion resistance; better wear-life and load-carrying capacity than air cured.
Ceramic: Glasses	500 to 1500	Good solvent and corrosion resistance; exceptional hardness; no vacuum outgassing; good radiation resistance; and high load capabilities.
Non-Ceramic: Silicates Phosphates	-300 to 1000	LOX compatible; no vacuum outgassing; good wear life; lack corrosion resistance.

Appendix C Substrate Materials

SUBSTRATE MATERIAL	RECOMMENDED PRETREATMENT	CRITICAL TEMP. °F*
ALUMINUM/ ALUMINUM ALLOYS	VAPOR DEGREASE ABRASIVE BLAST ANODIZE	275
COPPER/ COPPER ALLOYS	VAPOR DEGREASE ABRASIVE BLAST BRIGHT DIP OR BLACK OXIDE	400
CHROMIUM & NICKEL PLATING	VAPOR DEGREASE ABRASIVE BLAST	400
IRON	VAPOR DEGREASE GRIT BLAST PHOSPHATE	800
MAGNESIUM	VAPOR DEGREASE DICHROMATE TREATMENT	210
STAINLESS STEEL	VAPOR DEGREASE ABRASIVE BLAST PASSIVATE	400
STEEL/ STEEL ALLOYS	VAPOR DEGREASE GRIT BLAST PHOSPHATE	400
TITANIUM	SOLVENT WASH ABRASIVE BLAST ALKALINE ANODIZE	400

***Note:** Critical temperatures mark the onset of a decrease in strength and/or other mechanical properties of substrate materials.

Appendix D Test Methods for Solid Lubricants _____

TEST METHOD	TITLE OF METHOD
ASTM	
B-117	Salt Spray Testing
D-1005	Measurement of Dry Film Thickness of Organic Coatings
D-1212	Wet Film Thickness of Organic Coatings
D-1366	Reporting Particle Size Characteristics of Pigments
D-2247	Testing Water Resistance of Coatings in 100% Relative Humidity
D-2510	Adhesion of Solid Film Lubricants
D-2511	Thermal Shock Sensitivity of Solid Film Lubricants
D-2625	Wear-Life and Load-Carrying Capacity of Solid Film Lubricants (Falex Method)
D-2649	Corrosion Characteristics of Solid Film Lubricants
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