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3,223,626

CORROSION INHIBITING RESIN-BONDED SOLID FILM LUBRICANT

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The invention described herein may be manufactured and used by or for the Government for governmental purposes without payment to us of any royalties thereon.

The present invention relates to a solid film lubricant and more particularly to a resin-bonded solid film lubricant which combines good corrosion inhibiting properties with excellent wear life.

Resin-bonded solid film lubricants for use at temperatures up to 400 degrees Fahrenheit have been formulated for one of two main purposes; either (1) to provide long wear life or (2) to provide good corrosion protection. Two examples serve to illustrate these formulations. Example 1: Several commercial resin-bonded solid film lubricants, when applied over zinc phosphatized steel Falex test specimens and zinc phosphatized salt spray panels, were found to provide Falex wear lives within the range of 150–200 minutes at 50,000 p.s.i. load, but to provide only four hours corrosion protection in a 20 percent salt spray cabinet. Example 2: Other commercial resin-bonded solid film lubricants, when applied over zinc phosphatized steel Falex test specimens and zinc phosphatized salt spray panels, were found to provide Falex wear lives of approximately 60 minutes at a 50,000 p.s.i. load, but to provide corrosion protection for a period of 72 hours in a 20 percent salt spray cabinet.

An investigation also was made to determine the ability of a resin-bonded solid film lubricant containing graphite to prevent corrosion when applied over zinc phosphatized steel panels and exposed in a 20 percent salt spray cabinet. This study showed that zinc phosphatized steel panels coated with a resin-bonded solid film lubricant containing graphite corroded to a considerably greater extent than did similar zinc phosphatized steel panels with no lubricant coating exposed for the same length of time in a 20 percent salt spray cabinet. This phenomena of corrosion acceleration has also been corroborated by outdoor exposure tests. Although graphite is a common ingredient in resin-bonded solid film lubricants because of its beneficial synergistic effects when used with molybdenum disulfide, analysis indicates that corrosion acceleration is enhanced by the presence of graphite in the lubricant formulation.

Therefore, it is a primary object of the present invention to provide an improved resin-bonded solid film lubricant.

Another object of the present invention is to provide a resin-bonded solid film lubricant which has a relatively long wear life and, at the same time, excellent corrosion protection.

Still another object of the present invention is to provide an improved resin-bonded solid film lubricant without using graphite.

It is also an object of the present invention to provide a resin-bonded solid film lubricant which is relatively simple to prepare and which is highly effective as a lubricant.

In accordance with the present invention, a resin-bonded solid film lubricant composition is provided by admix-

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ing with an epoxy-phenolic resin system, quantities of antimony trioxide, molybdenum disulfide, magnesium bentonite, and p-dioxane.

The novel features of the present invention, as well as additional objects and advantages thereof, will be understood better from the following detailed description illustrated by way of two specific examples of compositions prepared in accordance with the present invention. These examples are given by way of illustration but should not be taken as limiting the present invention to the details thereof.

Example 1

	Percent by weight
Epoxy-phenolic resin system (35% solids) -----	¹ 30.2
Antimony trioxide, powdered, ACS grade -----	4.3
Molybdenum disulfide, natural 0.5 to 5.0 micron particle size -----	11.6
Modified magnesium bentonite -----	0.3
P-dioxane -----	53.6
	100.0

¹ Solids: 10.6.

An alternative lubricant may be prepared consisting of the following composition:

Example 2

	Percent by weight
Epoxy-phenolic resin system (35% solids) -----	¹ 36.6
Antimony trioxide, powdered, ACS grade -----	7.6
Molybdenum disulfide, natural 0.5 to 5.0 micron particle size -----	21.3
Modified magnesium bentonite -----	0.4
P-dioxane -----	34.1
	100.0

¹ Solids: 12.8.

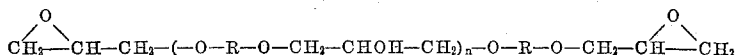
In the above identified examples, the epoxy-phenolic resin system which has been selected by preference is that known in the trade as "Epon Resin Formulation YP-100" prepared by Shell Chemical Corporation. The ingredients of this resin system are composed of 35 percent solids and 65 percent solvent taken from the following compositions:

	Percent by weight
Epon resin 1007 -----	73.1
General Electric resin 75108 -----	24.4
Silicone resin SR-82 -----	1.0
Phosphoric acid -----	1.5
Total solids -----	100.0
Ethyl alcohol -----	25.0
Methyl ethyl ketone -----	15.0
Toluene -----	50.0
Pine oil -----	10.0
Total solvent -----	100.0

In the above resin system, Epon resin 1007 is a commercial product made by Shell Chemical Company. Epon resin 1007 is a solid high molecular weight polyglycidyl ether of 2,2-bis (4-hydroxyphenyl) propane having the following properties:

Melting point ----- ° C.---	112–128
Mol weight -----	About 2,900
Epoxy value ----- Eq./100 g---	About 0.06

It is believed to have the general formula



wherein R is the portion of the 2,2-bis (4-hydroxyphenyl) propane molecule remaining after removal of the two OH groups and *n* is about 9. General Electric resin 75108 is a commercial product made by General Electric Company. It is a resin of the phenolic type with the following characteristics: Viscosity at 25 degrees centigrade, 2000-4000 centipoises; color, clear, dark brown; flash point, closed cup, over 200 degrees Fahrenheit; free water content, 0-2 percent; it is a mixture of the allyl ethers of mono-, di-, and trimethylol phenols, the last predominating (U.S. Patent No. 2,579,330, Miller, assignor to General Electric Co.). Silicone resin SR-82 is a commercial product made by General Electric Company. It is a resin of the silicone type with the following characteristics; specific gravity at 25 degrees centigrade, 1.19; color, maximum, 6 (Gardner); a hard solid compatible with a wide variety of compounds; soluble in xylene and toluene. Silicone resin SR-82 is methylphenylpolysiloxane characterized by repeating units of the general formula



wherein R are methyl and phenyl radicals (U.S. Patent No. 2,258,222, Rochow, assignor to General Electric Co.).

Antimony trioxide is an oxide from the class of inorganic metallic oxides. While other metallic oxides might be used, antimony trioxide is preferred since other metallic oxides from this class are not found to be as effective. The modified magnesium bentonite is that known as Bentonite 38, made by Baroid Division of National Lead Company. This is a finely powdered material made by replacing the inorganic cations of a clay mineral lattice with organic cations. The solvent p-dioxane is technically known as 1,4 diethylene dioxide, sometimes called para dioxane. Although other solvents can be used, such as a mixture of equal parts of diacetone and xylene, the results obtained are not considered to be as satisfactory. Therefore, it is preferred that p-dioxane be used as the solvent.

The ingredients in the formulations set forth in Examples 1 and 2 above were prepared by adding them in the following order to a one gallon friction lid can containing approximately 20 one-half inch diameter steel balls:

- (1) Epoxy-phenolic resin system;
- (2) Antimony trioxide;
- (3) Molybdenum disulfide;
- (4) Modified magnesium bentonite; and
- (5) P-dioxane

There was substantially no time lag between the addition of each ingredient. The can was sealed, shaken by hand for approximately one minute and then rolled for four hours at room temperature. The formulation is prepared at room temperature and no heating is required.

The formulations outlined above were applied over zinc phosphatized steel test specimens as coatings by dipping. It is always considered best practice to steel grit blast or sand blast steel surfaces prior to zinc phosphatizing. All test specimens were steel grit blasted prior to zinc phosphatizing. Inasmuch as the formulation contains a relatively high percentage of solvent, it was necessary to dip each specimen three times in order to obtain a coating thickness of 0.0004 to 0.0007 inch. The dipping procedure was accomplished by suspending the specimen in the lubricant for a period of 5 seconds for each successive dip, removing it after each dip and allowing it to dry for ten minutes after each of the first two dips before proceeding with the next dip. After the final dip, the specimen was allowed to dry for one half hour followed by oven curing to bake the resin at 400 degrees Fahrenheit for one hour.

In the case of the lubricant prepared in accordance with the formulation set forth in Example 2 above, the procedure for preparation of test specimens was similar to that for the lubricant prepared as in Example 1. However, since the amount of solvent was considerably less, the specimens only required a single dip for five seconds to provide a similar coating thickness after which they were removed and air dried for approximately one hour. The dried specimens were baked in an oven at 400 degrees Fahrenheit for one hour.

The coatings provided by the above described formulations were tested and found to give maximum wear life and, at the same time, excellent corrosion protection when applied to zinc phosphatized steel as well as anodized and sealed aluminum. For example, the test specimens coated with lubricant in accordance with Example 1 above, consisted of American Iron and Steel Institute, also referred to herein as AISI, C1020 steel panels 1/8 x 2 x 3 inches for the salt spray cabinet exposure and standard Falex test specimens for the wear test. The Falex pins were made from AISI 3135 steel hardened to Rockwell B87-90. The V-blocks were made from AISI 1137 steel with a hardness of Rockwell C20. These specimens were found to provide 72 hours corrosion protection in the 20 percent salt spray cabinet with a Falex wear life of 337 minutes. The kinetic coefficient of friction was 0.01. Similar specimens coated with lubricant in accordance with Example 2 above provided 24 hours corrosion protection in the 20 percent salt spray cabinet with a Falex wear life 365 minutes. The kinetic coefficient of friction in this case was 0.01.

It will be appreciated that certain changes and variations may be made in the specific formulations described herein without departing from the scope and the product provided by the present invention. Depending upon the particular application of the lubricant, the composition is susceptible of variation within the following ranges:

Percentages by weight	
Epoxy-phenolic resin system (35% solids) -----	20 to 50 (solids: 7 to 17.5).
Antimony trioxide, powdered, ACS grade -----	4.3 to 30.
Molybdenum disulfide, natural ---	11.6 to 70.
Modified magnesium bentonite ---	2 to 20 grams per gallon of finished product.
P-dioxane -----	Sufficient to produce a final percentage of solids ranging from 20 to 50 percent.

¹ Solids: 7 to 17.5.

Although the above constituents may be varied within the ranges indicated, it is recommended that care should be exercised in the ratio of antimony trioxide to molybdenum disulfide since this ratio is critical from the point of view of Falex wear life.

What is claimed is:

1. A graphite-free film lubricant composition consisting essentially of a solution of

Parts by weight, about—

A mixture of an epoxy resin being a high molecular weight polyglycidyl ether of 2,2-bis(4-hydroxyphenyl)-propane, and of a methylol-phenolic resin being an allyl ether of methylol-substituted phenol ---	17 to 17.5
Antimony trioxide -----	4.3 to 30
Molybdenum disulfide -----	11.6 to 70

¹ Solids.

in a solvent comprising p-dioxane, said solution having a solids content of about 20 to 50 percent.

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2. A graphite-free film lubricant composition consisting essentially of a solution of

Parts by weight, about—

A mixture of an epoxy resin being a high molecular weight polyglycidyl ether of 2,2-bis-(4-hydroxy-phenyl)-propane, and of a methylol-phenolic resin being an allyl ether of methylol-substituted phenol	5
Antimony trioxide	¹ 10.6
Molybdenum disulfide	4.3
	11.6 10

¹ Solids.

in a solvent comprising p-dioxane, said solution having a solids content of about 20 to 50 percent.

3. A graphite-free film lubricant composition consisting essentially of a solution of

Parts by weight, about—

A mixture of an epoxy resin being a high molecular weight polyglycidyl ether of 2,2-bis-(4-hydroxy-phenyl)-propane, and of a methylol-phenolic resin being an allyl ether of methylol-substituted phenol	20
Antimony trioxide	¹ 12.8
Molybdenum disulfide	7.6
	21.3 25

¹ Solids.

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in a solvent comprising p-dioxane, said solution having a solids content of about 20 to 50 percent.

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