United States Patent Office
3,288,710
Patented Nov. 29, 1966

3,288,710
HIGH LOAD CAPACITY DRY FILM LUBRICANTS

Rhynie F. Hollitz, Midland, Mich., assignor to Dow Corning Silicones Limited, Toronto, Ontario, Canada, a corporation of Canada

No Drawing. Filed Feb. 21, 1964, Ser. No. 346,409
11 Claims. (Cl. 252—25)

This application relates to new dry film lubricants for metal that have the capability of withstanding heavy loads without rupture of the film, which causes loss of lubrication and final disruption of the film.

Dry film lubricants are commonly used to give metal surfaces a permanent, dry coating that serves as a lubricant, in that it greatly reduces the coefficient of friction of the metal contacting surfaces. In many applications dry film lubricants eliminate the need for the oiling of bearings, sliding surfaces, etc.

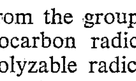
Dry film lubricants generally consist of a solid lubricant, such as molybdenum disulfide or graphite, plus a resin that is capable of forming a cured film. The mixture of solid lubricant and resin is applied as a film over the metal surface that is to be lubricated, and the film is cured by air drying, baking, etc. The result of this is a dry lubricating film; the solid lubricant does the lubricating, and it is held in place by the resin film, making the lubricating action long lasting.

Presently available commercial dry film lubricants, however, are limited in their capability to carry heavy loads without rupture of the dry film, which causes a sudden and potentially disastrous increase in friction to the point where the two metal surfaces can weld together. This is most likely to happen when the bearing or sliding surface is starting up its motion. Many dry films which otherwise function well can be ruptured by the surge of frictional force that accompanies start up.

It is an object of this invention to create new dry film lubricants that function well at high loads, many of which exceed all presently-sold dry film lubricants in load-bearing capacity. These dry film lubricants also give superior moisture protection of the lubricating composition which they contain. For lubricating compositions such as MoS_2, this can be a big advantage, eliminating possible formation of acids.

The compositions of this invention are described in the following manner:

A lubricating composition consisting essentially of (1) from 40 to 85 weight percent of a dry lubricant, and (2) from 15 to 60 weight percent of a resinous composition consisting of an epoxy resin which is a condensation product (a) from 40 to 100% by weight of an aromatic epoxy resin; and, (b) from 0 to 60% by weight of an organosilicon compound of the formula



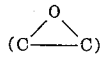
$$R_nSi(R'_m)O_{\frac{4-m-n}{2}}$$

where R is selected from the group consisting of monovalent aromatic hydrocarbon radicals and lower alkyl radicals, R′ is a hydrolyzable radical, *n* has an average value of 0.8 to 3, and *m* has an average value of 0.1 to 3.2, the sum of *n*+*m* being not greater than 4.

As can be seen from the above description, the composition of this invention is operative as a dry film lubricant without any silicone ingredient, but it is generally preferred for ingredient (2) to contain from 5 to 40% by weight of silicone. The silicone appears to enhance the load-bearing ability and the heat stability of the film. Example 5, however, shows a composition that contains no silicone, but works very well.

Ingredient (1) can be any dry lubricant such as molybdenum disulfide, graphite, antimony trioxide, titanium disulfide, tungsten disulfide, bismuth, lead sulfide, lead oxide, polytetrafluoroethylene, magnesium oxide, boron nitride, calcium fluoride, zirconium disulfide, colloidal silver, etc. Suitable combinations of dry lubricants can also be used, such as the combination of graphite and molybdenum disulfide, and combinations of calcium fluoride, molybdenum disulfide, and boron nitride.

By the term "aromatic epoxide resin," as used in the description of ingredient (2) it is meant those resins that are produced by the reaction of polyhydric phenols with either polyfunctional halohydrins or polyepoxides or mixtures thereof to form complex reaction products containing terminal epoxide groups



By "consisting essentially of," it is implied that other ingredients can be present in the compositions of this invention. Examples of these are solvents to plasticize or dissolve the uncured composition, and modifiers for the aromatic epoxy resin such as non-aromatic epoxy resins to make the cured composition softer, and fatty acids and mixtures thereof such as octanoic acid, stearic acid, linseed oil acid, tung oil acid, castor oil acid, etc., which fatty acids tend to enhance the cure, and the lubricity of the compositions of this invention at low loads.

The polyhydric phenols used in making the epoxide resins employed herein include any of the phenols containing two or more phenolic hydroxyl groups, which can be on one phenyl ring as in resorcinol, or in different, fused ring systems, as in 1,5-dihydroxy naphthalene, or in different ring systems which are attached by chains composed of one or more atoms, in which case the chains should be free from elements which interfere with the reaction of the polyepoxides with the phenolic hydroxyl groups.

The phenolic nuclei can contain substituents (such as ring substituted halogen atoms) providing that they also do not interfere with the aforesaid reaction.

Illustrative of polyhydric phenols which can be used in making the epoxide resin copolymers are monophenyl phenols such as resorcinol, hydroquinone, catechol, phloroglucinol; polyphenyl phenols such as bisphenol(p,p-dihydroxydiphenyl dimethylmethane), p,p-dihydroxy benzophenone, p,p-dihydroxydiphenyl, p,p-dihydroxydibenzyl, bis(4-hydroxyphenyl)sulfone, 2,2-dihydroxy-1,1-dinaphthylmethane, polyhydroxynaphthalenes and anthracenes, o,p,o,p-tetrahydroxydiphenyldimethylmethane, etc.

Also operative in this invention are polyhydric phenols which are condensation products of simpler polyhydric phenols with dichlorides such as dichlorodiethyl ether and dichlorobutene and are assumed to have one of the following general formulae:

$$HOR[OCH_2CH_2OCH_2CH_2OR]_nOH$$

and/or

$$HOR[OCH_2CH{=}CHCH_2OR]_nOH$$

wherein R is the residue from the phenol and *n* is at least 1. These polymeric condensation products consist of the dihydric phenol residues united or joined by and through the residues from the organic dichlorides.

A special case of interest and operative herein involves the condensation of complex polyhydric phenols with dibasic acids. For example, the condensation of adipic acid with a polyhydric phenol produces a polymer of the general formula

$$HOR[O\overset{O}{\overset{\|}{C}}-(CH_2)_4\overset{O}{\overset{\|}{C}}-OR]_nOH$$

in which R is the residue from the phenol and *n* is at least 1.

One of the best known and preferred of the polyhydric phenols operative herein is bis-phenol-(p,p'-dihydroxydiphenyldimethyl methane).

The polyepoxides which are operative herein as reactants with the polyhydric phenols contain two or more epoxide groups. The simplest diepoxides contain at least 4 carbon atoms such as 1,2-epoxy-3,4-epoxy butane. The epoxy groups can be separated from each other by ether groups or linkages as in the case of bis-(2-3-epoxypropyl)-ether and bis-(2,3-epoxy-2-methylpropyl)ether. Also operative herein are the more complex polyepoxides such as those prepared by reacting 2 or more mols of a diepoxide with 1 mol of dihydric phenol, or 3 or more mols of a diepoxide with 1 mol of trihydric phenol, etc. Polyepoxides derived from polyhydric alcohols such as mannitol, sorbitol, erythritol or polyallyl alcohol can also be used. The polyepoxy compounds used herein can have varying structures and can be of complex structure so long as they do not contain groups which interfere with the reaction between the epoxide groups and the phenolic hydroxyl groups. The polyepoxide reactants employed herein should be substantially free from reactive groups other than epoxide and aliphatic hydroxyl groups.

Simple diepoxides of a high degree of purity can be prepared and obtained by fractional distillation to separate them from byproducts formed during their manufacture. For example, bis-(2,3-epoxypropyl)ether or diglycid ether can be produced and separated by fractional distillation to give products of high purity. More complex polyepoxides of higher molecular weight are generally difficult to isolate by fractional distillation, but they can be employed herein after purification to remove objectionable inorganic impurities and catalysts such as caustic alkali. The high molecular weight diepoxide or polyepoxide with admixed byproducts such as monoepoxides is operative herein. Particularly well known and useful herein are the reactive products of, for example, epichlorohydrin with a polyhydric alcohol. A specific example of this reaction involves 1 mol of a trihydric alcohol reacted with 3 mols of epichlorohydrin and a catalyst. The epoxide group of the epichlorohydrin reacts with a hydroxyl group of the alcohol and subsequent treatment removes chlorine from the reaction product thus producing the desired polyepoxide. Such polyepoxides can contain less than 3 epoxy groups per molecule even though 3 mols of epichlorohydrin are reacted with 1 mol of a trihydric alcohol. Complex side reactions apparently take place with the resulting production of other compositions containing free hydroxyl groups or cyclic ring compounds or polymeric compounds which can be present in the resulting product. Nevertheless, such products can be employed as polyepoxides for reaction with polyhydric phenols to form the resinous epoxides of this invention.

The polyepoxides can contain varying small amounts of admixed monoepoxides. To the extent that monoepoxides are present they will react with the polyhydric phenols to form terminal groups or residues containing hydroxyl groups and to the extent that such terminal hydroxyl groups are present the complex polyepoxide compositions will contain complex epoxy-hydroxyl compounds containing both terminal epoxide-containing residues and terminal hydroxyl-containing residues. The presence of monoepoxides or of monoepoxy-hydroxyl compounds does not interfere with the desired reaction provided a sufficient amount of polyepoxides is present to serve as polyfunctional reactants with polyhydric phenols.

The reaction conditions and proportions of reactants to be employed and obvious variations of the reaction as well as alternative reactions are set forth in full detail in United States Patent 2,592,560 issued April 15, 1952.

Alternatively, the polyhydric phenol can be reacted with a polyfunctional chlorohydrin such as monochlorohydrins, for example, epichlorohydrin, dichlorohydrins, for example, glycerol dichlorohydrin, bis-(3-chloro, 2-hydroxypropyl)ether, 1,4-dichloro-2,3-dihydroxy butane, 2-methyl-2-hydroxy-1,3-dichloropropane, bis(3-chloro, 2-methyl, 2-hydroxypropyl)ether, and other mono and dichlorohydrins derived from aliphatic olefins, mannitol, sorbitol and other alcohols. In short, the term "polyfunctional chlorohydrin" as employed herein includes all compounds which contain at least one epoxide and at least one chlorine atom in the molecule and all compounds containing a chlorine atom and an OH group on adjacent carbon atoms and at least one other chlorine atom in the molecule. It is preferred that the chlorohydrin be substantially free of other functional groups. Epichlorohydrin and/or glycerol dichlorohydrin are particularly useful in this invention.

The proportions of reactants as well as reaction conditions and variations and alternative procedures involved in the polyhydric phenol-polyfunctional chlorohydrin reaction are well known and are set forth in detail in U.S. Patents 2,615,007 and 2,615,008.

The uncondensed silicone portion of ingredients (2) is a silane or silanol of the formula $R_nSiR'_{4-n}$, or a siloxane containing R and R' groups.

R can be any monovalent aromatic hydrocarbon radical, such as phenyl, tolyl, biphenyl, naphthyl, n,n-diethylphenyl, anthracenyl, chrysenyl, etc.; or any lower alkyl radical such as methyl, ethyl, hexyl, isobutyl, etc.

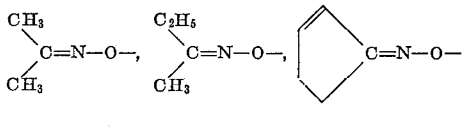
R' can be —OH or any hydrolyzable group; e.g. halogens such as fluorine, chlorine, and bromine; alkoxy groups such as methoxy, ethoxy, isopropoxy, decoxy, etc.; polyalkoxy such as beta-ethoxyethoxy, beta-methoxyethoxy, and $CH_3OC_2H_4OC_2H_4O—$; haloalkoxy groups such as beta-chloroethoxy, 3,3-difluoropropoxy,

$$CCl_3CH_2OCH_2CH_2O—$$

etc; acyloxy groups such as acetate, propionate, isobutyrate and octoate; haloacyloxy such as beta-chloroacetate, 3,3-difluoropropionate, etc.; the isocyanate group; amino groups such as $—NH_2$, $—NHCH_3$, $—NHC_2H_5$,

$$—NHC_6H_{11},\ —N(CH_3)_2$$

and groups of the ketoxime class such as



$$(CH_3)_2C=N—O—,\quad (C_2H_5)(CH_3)C=N—O—,$$

etc.

When the hydrolyzable group is such that reaction with the OH groups in the epoxide will produce strongly acidic or amine byproducts, the condensation should be carried out at low temperatures and under anhydrous conditions in order to minimize reaction of the HR' byproduct with the oxirane groups on the epoxide resin.

The preferred hydrolyzable groups are alkoxy.

The ingredients that make up ingredient (2) can be cocondensed by heating in a manner that is well known to the art (e.g. see Canadian Patent 662,941). The condensation can be carried out at room temperature in those cases where the R' group is very reactive.

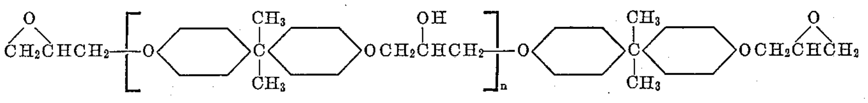
Ingredients (1) and (2) are then mixed intimately, the best way being to dissolve them in the presence of a solvent, and apply them to the metal surface for curing.

The compositions of this invention cure in the range of 100° to 200° C. Upon curing they harden into cured resinous materials in which the dry lubricant is entrapped. The compositions form good films on curing, which expose the dry lubricant on the surface of the film, but prevent the dry lubricant from migrating when the film is under great pressure. These dry film lubricants also have the added advantage of preserving the dry lubricant from the effects of moisture.

The following examples are illustrative only and should not be construed as limiting the invention, which is properly delineated in the appended claims.

Example 1

A composition was prepared consisting of 70% by weight MoS_2, 7% graphite, and 23% of a composition consisting of the following: The condensation product of (a) 15 parts of the hydrolyzate of 70 mol percent monophenyltrichlorosilane and 30 mol percent monopropyltrichlorosilane, hydrolyzed in one-half of the molar weight of the silanes of methyl isobutyl Cellosolve and excess toluene; and (b) 45 parts of



where n equals approximately 2, mixed with 20 parts of xylene, 20 parts of beta-ethoxyethyl acetate, and 1 part of 2-ethylhexylic acid.

This composition was milled in a colloid mill, and diluted with toluene until it was sprayable.

This composition was sprayed on two vapor-blasted steel V blocks and a pin that fits into the V block, and cured for one hour at 300° F.

The pins and blocks used for the tests were one-quarter inch diameter and "liquid honed" using 50% No. 625 and 50% No. 1250 abrasives, to provide consistent surface finishes with maximum surface area. Prior to spraying the dry films, pins and blocks were washed in acetone and were not touched with the hands until the curing cycles and testing were completed. The pins are of SAE 3135 steel, with a Rockwell "B" hardness of 87–90, and the V blocks of A151–1137 steel with a Rockwell "C" hardness of 20 and a block angle of 96°.

The frictional characteristics of the pin rotating in the blocks were determined by means of a Falex lubricant testing machine, using a "step test." By this technique the load on the pin and block is increased stepwise at periodic intervals, the torque of the rotating pin being constantly measured.

The results are as follows:

Total Load (lb.)	Torque at beginning of period (lb./in.)	Torque at end of period (lb./in.)	Duration of period of load (min.)
250	5	1	3
500	4.5	4.5	1
750	7	7	1
1,000	8	8	1
1,250	10	9	1
1,500	11	10.5	1
1,750	11.5	11.5	1
2,000	12	12	1
2,250	12	12	1
2,500	12	12	1
2,750	12	12.5	1
3,000	13	13.5	1
3,250	13	13	1
3,500	13.5	13.5	1
3,750	13.5	14.0	1
4,000	14	14	1
4,250	14.5	14.5	1
4,500	15	30	(1)

[1] Failure after 55 seconds.

Example 2

The composition of Example 1 was reformulated with the graphite ingredient being replaced with 7% of calcium fluoride.

The composition was run through a colloid mill, and to it was added enough of a mixture of 50% ethylene glycol and 50% xylene to make the composition sprayable.

The composition was sprayed on a block and pin set, cured, and step-tested in the manner of Example 1.

The results follow:

Total Load (lb.)	Torque at beginning of period (lb./in.)	Torque at end of period (lb./in.)	Duration of period of load (min.)
250	3	1.5	3
500	4	4	1
750	5.5	5.5	1
1,000	7.5	7.5	1
1,250	8.5	8	1
1,500	9	9	1
1,750	10.5	9.5	1
2,000	11	10	1
2,250	11.5	11	1
2,500	12	11	1
2,750	11	11	1
3,000	12	11	1
3,250	12	11.5	1
3,500	12.5	11.5	1
3,750	12	12	1
4,000	12.5	13	1
4,250	13	13.5	1
4,500	15.5	17.5	[1] 2

[1] Not maintaining load.

Example 3

The composition of Example 1 was reformulated, replacing the graphite with 7% of boron nitride.

The composition was run through a colloid mill, and to it was added enough of a mixture of 50% ethylene glycol and 50% xylene to make the composition sprayable.

The composition was sprayed on a block and pin set, cured, and step-tested in the manner of Example 1, except that the block and pin set had received the commercially-available phosphate-manganese anticorrosion treatment.

The treated pins performed well in the step-test, low torque being recorded. At the final load of 4500 pounds, which was sustained for one minute, the torque at the beginning of the period was 11 pound inches and at the end of the period was 15 pound inches, which was the highest torque recorded in the experiment.

Example 4

The composition of Example 1 was reformulated, replacing the 70% of MoS_2 and the 7% of graphite with 60% of MoS_2, 10% of antimony trioxide, and 7% of zirconium sulfide.

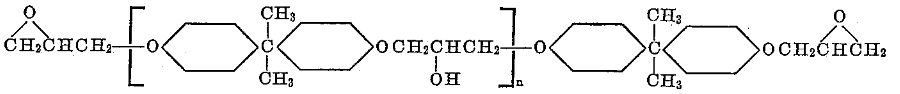
The composition was run through a colloid mill, and to it was added enough of a mixture of 50% ethylene glycol and 50% xylene to make the composition sprayable.

The composition was sprayed on four block and pin sets, cured, and tested in the manner of Example 1, except that the block and pin sets had received the commercially-available phosphate-manganese anticorrosion treatment, and except that the test runs were at a constant load of 1000 pounds.

The four block and pin sets endured an average of 2 hours and 9 minutes of continuous operation at the 1000 pound load before failure of the dry film lubricant. The first block and pin set failed after two hours of operation, and the last set failed after 2 hours and 14 minutes of operation.

Example 5

A composition was prepared consisting of 70% by weight MoS_2, 7% graphite, and 23% of



where *n* equals approximately 2.

A small amount of a solvent which was a mixture of 50% methyl isobutyl ketone and 50% toluene was added to plasticize the composition, which was then run through a colloid mill.

More of the above solvent was then added so as to make the composition sprayable.

It was then sprayed on a pin and block set, cured, and step-tested in the manner of Example 1.

The results were as follows:

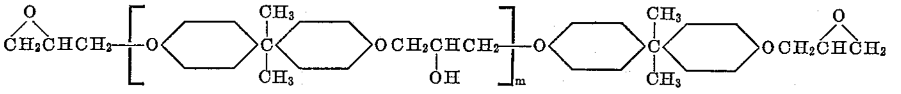
Total Load (lb.)	Torque at beginning of period (lb./in.)	Torque at end of period (lb./in.)	Duration of period of load (min.)
250	3.5	1.5	3
500	2.5	2.5	1
750	3.0	2.5	1
1,000	3.5	3.5	1
1,250	4.5	4.5	1
1,500	5.5	5.0	1
1,750	6.0	6.0	1
2,000	6.5	6.5	1
2,250	7.0	7.0	1
2,500	8.0	8.5	1
2,750	8.5	9.5	1
3,000	9.5	10.5	1
3,250	14.5	9.5	1
3,500	16.5	34+	<1

Example 6

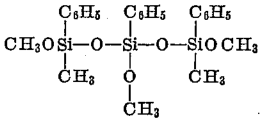
Eighteen commercial dry film lubricants were applied to V blocks and pins by methods in accordance with the manufacturers' recommendations, and tested on the Falex machine by the "step test" as described in Example 1. The best of the eighteen compositions failed at a load of 3250 pounds, i.e. the point where a rapid rise of torque to the point of welding of the pin to the blocks were observed. The two second best compositions failed at a load of 2750 pounds. The approximate mean failing load was 1750 pounds.

Example 7

When 40 parts by weight of magnesium oxide are intimately mixed in toluene with 60 parts by weight of a cocondensate of 95 weight percent of



where *m* equals approximately 100; and 5 weight percent of a silicone of the average formula:



$$\mathrm{CH_3O\overset{C_6H_5}{\underset{CH_3}{Si}}-O-\overset{C_6H_5}{\underset{\underset{CH_3}{O}}{Si}}-O-\overset{C_6H_5}{\underset{CH_3}{Si}}OCH_3}$$

a material is formed that cures on metal as a film by heating and imparts good lubricating qualities to the metal surface.

Example 8

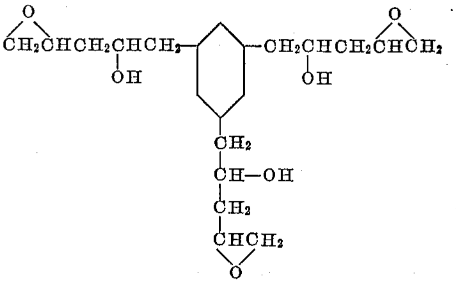
When 50 parts by weight of titanium disulfide are intimately mixed in acetone with 50 parts by weight of a cocondensate of (a) 90 weight percent of the condensation product of equal molar quantities of resorcinol and 1,2-epoxy-3,4-epoxy butane; and (b) 10 weight percent of phenylethylsilanediol, a material is formed that cures on metal as a film by heating, and imparts lubricating qualities of the metal surface.

Example 9

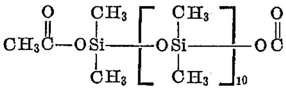
When 60 parts by weight of polytetrafluoroethylene are intimately mixed in diethyl ether with 40 parts by weight of a cocondensate of (a) 70 weight percent of the condensation product of 40 mol percent of phloroglucinol and 60 mol percent of bis(2,3-epoxypropyl)ether; and (b) 23 weight percent of tolyltriethoxysilane, 2 weight percent of trimethylchlorosilane, and 5 weight percent of ethylsilicate, a material is formed that cures on metal as a film upon heating, and imparts lubricating qualities of the metal surface.

Example 10

When 70 parts by weight of lead sulfide are intimately mixed with 30 parts of a cocondensate of (a) 60 weight percent of the condensation product of 40 mol percent of 1,3,7-trihydroxynaphthylene, 10 mol per cent of



40 mol percent of bis(2,3-epoxy-2-methylpropyl)ether, and 10 mol percent of 1,2-epoxypropane; and (b) 40 weight percent of

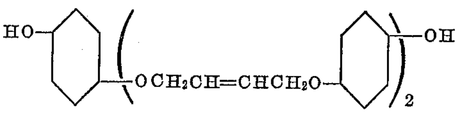


$$\mathrm{CH_3\overset{O}{\overset{\|}{C}}-O\overset{CH_3}{\underset{CH_3}{Si}}\left[-O\overset{CH_3}{\underset{CH_3}{Si}}-\right]_{10}-O\overset{O}{\overset{\|}{C}}CH_3}$$

a material is formed that cures on metal as a film upon heating, and imparts lubricating qualities to the metal surface.

Example 11

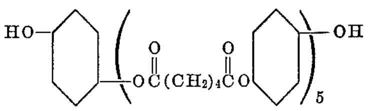
When 80 parts by weight of colloidal silver are intimately mixed in benzene with 20 parts of a composition containing 50 weight percent of the condensation product of equimolar quantities of



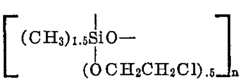
and glycerol dichlorohydrin; and 50 weight percent of anthracenylhexyl tetra-beta-ethoxyethoxydisiloxane, a material is formed that cures on metal as a film on heating, and imparts lubricating qualities to the metal surface.

Example 12

When 85 parts by weight of finely divided bismuth are intimately mixed in cyclohexane with 15 parts by weight of a composition consisting of 40 weight percent the condensation product of equimolar portions of



and 1,4-dichloro-2,3-dihydroxy butane, and 60 weight percent of a polymer of the average formula

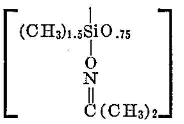


$$\left[(CH_3)_{1.5}\underset{(OCH_2CH_2Cl)_{.5}}{\overset{|}{Si}}O-\right]_n$$

a material is formed that cures on metal as a film on heating, and imparts lubricating qualities to the metal surface.

Example 13

When the experiment of Example 11 is repeated substituting

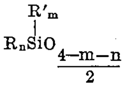


$$\left[(CH_3)_{1.5}\underset{\substack{|\\O\\|\\N\\\|\\C(CH_3)_2}}{\overset{|}{Si}}O_{.75}\right]$$

for the silicone ingredient of Example 11, a material is formed that cures on metal as a film on heating, and imparts lubricating qualities to the metal surface.

That which is claimed is:

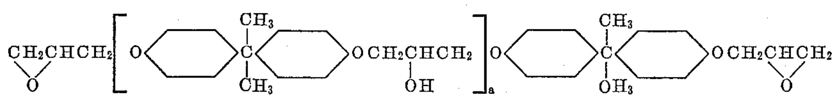
1. A dry film lubricant consisting essentially of
   (1) from 50 to 85 weight percent of a dry lubricant, and
   (2) from 15 to 50 weight percent of a resinous composition consisting essentially of a silicone-modified epoxy resin which is a condensation product of the following units:
      (a) from 60 to 95% by weight of an aromatic epoxy resin; and,
      (b) from 5 to 40% by weight of an organosilicon compound of the formula



$$R_n\overset{R'_m}{\overset{|}{Si}}O_{\frac{4-m-n}{2}}$$

where
R is selected from the group consisting of monovalent aromatic hydrocarbon radicals and lower alkyl radicals,
R' is a hydrolyzable radical,
n has an average value of 0.8 to 2, and
m has an average value of 0.1 to 3.2, the sum of
m and *n* being not greater than 4.

2. The lubricant of claim 1 where (*a*) is



where *a* is an integer of 0 through 100.

3. The lubricant of claim 1 where R consists of phenyl and propyl units.

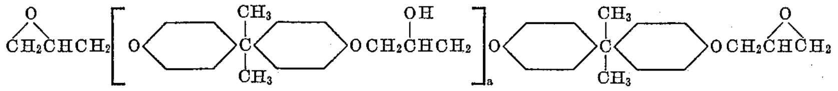
4. The lubricant of claim 1 where R consists of phenyl and methyl units.

5. The lubricant of claim 1 where the dry lubricant is selected from the group consisting of CaF_2, MoS_2, BN, and mixtures thereof.

6. The lubricant of claim 1 where the dry lubricant is a mixture of MoS_2 and graphite.

7. The lubricant of claim 1 where R' is methoxy.

8. A dry film lubricant consisting essentially of 70 weight percent of MoS_2, 7% of graphite; 13.8% of a composition consisting of 75 weight percent of



where *a* has an average value of 2, and 25 weight percent of the hydrolyzate of 70 mol percent of monophenyltrichlorosilane and 30 mol percent of monopropyltrichlorosilane; and 9.2% by weight of solvent.

9. The lubricant of claim 8 where the graphite ingredient is replaced by CaF_2.

10. The lubricant of claim 8 where the graphite ingredient is replaced by BN.

11. The lubricant of claim 8 where the MoS_2 and graphite ingredients are replaced with 60% by weight of MoS_2, 10% antimony trioxide, and 7% of zirconium sulfide.

References Cited by the Examiner

UNITED STATES PATENTS

2,592,560	4/1952	Greenlee	260—47
2,615,007	10/1952	Greenlee	260—47
2,615,008	10/1952	Greenlee	260—47
3,079,338	2/1963	Caubet	252—25 X

FOREIGN PATENTS

662,941	5/1963	Canada

OTHER REFERENCES

"Bonded Coatings Lubricate Metal Parts," by Di Sapio in Product Engineering, Sept. 5, 1960, pp. 48–53.

"Recent Developments in Lubricants," by Cosgrove et al. in Battelle Technical Review, April 1961, pp. 1–7.

"Manufacture and Application of Lubricating Greases," by Boner, Reinhold Pub. Corp., New York, 1954, p. 786.

DANIEL E. WYMAN, *Primary Examiner.*

I. VAUGHN, *Assistant Examiner.*

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

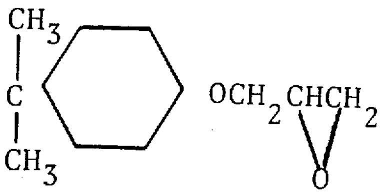
Patent No. 3,288,710

November 29, 1966

Rhynie F. Hollitz

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Columns 9 and 10, lines 5 to 8, the right-hand portion of the formula should appear as shown below instead of as in the patent:



Signed and sealed this 24th day of October 1967.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents