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GREASE COMPOSITION

Samuel Meghir, Chicago, Ill., assignor to Standard Oil Company, Chicago, Ill., a corporation of Indiana

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ABSTRACT OF THE DISCLOSURE

Lubricant greases especially suitable for use at elevated temperatures of about 400° F. to about 600° F., and higher, are made by thickening a normally liquid lubricant with a finely divided silica which has been surface-treated with a hexa(C_{1-4} alkyl) disilazane, e.g. hexamethyldisilazane, or with a fluorosilane derivative prepared by reacting dimethyldichlorosilane with a perfluoroalcohol, e.g. a C_{11}-fluoroalcohol.

The invention herein described was made in the course of or under a contract with the Department of the Air Force.

BACKGROUND OF THE INVENTION

The adequate and proper lubrication of certain equipment, particularly certain types of military planes, requires grease lubricants that meet a variety of conditions ranging from atmospheric to space environments. Hence, a lubricant grease to be satisfactory must be effective at temperatures as high as about 400 to 600° F. and higher, and under high rotational speeds.

Finely divided silica is itself stable at high temperatures and is known to thicken fluids; U.S. 2,554,222, issued May 22, 1951, to F. H. Stross, discloses the use of silica gel as a grease thickener. However, grease thickened with silica is unstable at high temperatures, i.e. at 400° F. or higher. Surface treatments of silica have been used to increase compatability with lubricant fluids and to improve other properties. Thus silica has been treated with alcohol to form ester linkages. Such surface-treated silicas have been referred to as estersils. However, such estersils are unstable when subjected to bearing tests at 600° F.

SUMMARY

The object of the present invention is to provide grease lubricants thermally stable at temperatures in the range of from about 400° F. to about 600° F. and higher, and under high rotational speeds.

Such thermally stable grease lubricants are obtained, in accordance with this invention, by thickening a thermally stable lubricating oil to the desired consistency or penetration by admixing with such lubricating oil a finely divided silica which has been surface-treated with (a) a hexaalkyldisilazane of the general formula

$$(R)_3SiNHSi(R)_3$$

in which R represents the same or different C_{1-4} alkyl radicals, or (b) with a fluorosilane derivative prepared by reacting dimethyldichloro silane with a C_3 to C_{17} perfluoroalcohol in equimolar amounts.

In preparing the lubricant grease, the surface-treated silica, in sufficient quantity, e.g. about 2–40% of the final grease composition, is added to the oleaginous lubricant vehicle and stirred at about room temperature until the mixture is unifrom and then milled. The milled mixture is heated to about 400° F. to 500° F. and maintained at such temperature for a period of about 0.1 to 10 hours; and thereafter cooled to about room or ambient temperature to form a homogenous grease product.

The oleaginous vehicle can be any normally liquid lubricant such as, mineral lubricating oils; synthetic hydrocarbon oils; synthetic lubricating oils, e.g. polyalkylene glycols and their derivatives, or high molecular weight esters of dicarboxylic acids; silicone polymer oils, i.e. silicone oils within the lubricating oil range; polyfluoro derivatives of organic compounds, for example perfluoro polymeric fluids, such as trifluorochloroethylene polymers; dibasic acid esters of $H(CF_2)_nCH_2OH$, trifluorovinyl chloride polymers etc. When the grease product is to be employed under elevated temperatures, e.g. above about 400° F., oleaginous lubricant vehicles which are thermally stable at the temperatures to be encountered should be used. For such uses, silicone polymer oils and perfluoro polymeric fluids, such as the product marketed as "PR 143" by Du Pont, are preferred.

Illustrative of hexaalkyldisilazanes which may be employed are hexamethyldisilazane, hexaethyldisilazane, hexabutyldisilazane, trimethyltripropyldisilazane, triethyltrimethyldisilazane etc.

The perfluoroalcohol reacted with the dimethyldichlorosilane has the general formula $H(CF_2CF_2)_xCH_2OH$, wherein x is an integer of 1 to about 8. In preparing the fluorosilane derivative, the perfluoroalcohol is reacted at room temperature with the dimethyldichlorosilane in equimolar amounts.

In the preparation of the surface-treated silica, the hexaalkyl disilazane or the fluorosilane derivative is reacted with the finely divided silica for about two hours in a reflux system in toluene suspension at the toluene boiling point temperature under a slow stream of nitrogen. The toluene is subsequently distilled off and the surface-treated silica dried at 400° F. under slight vacuum. Some boric acid has been found to be an effective stabilizer for silica-thickened greases, it is therefore advantageous to add about 2%, based on the silica, to the mixture of silica and the hexaalkylidisilazane or the fluorosilane derivative.

The finely divided silicas, which are surface-treated with either the hexaalkyldisilazane or with the above described fluorosilane, have particle sizes predominantly in the 0.005–.020 micron range, with surface areas (BET nitrogen adsorption isotherm) of about 100–350 square meters per gram. Such silicas are prepared by various known techniques, including low temperature precipitation from aqueous solutions of soluble silicates, high temperature i.e. 100° C., vapor phase hydrolysis of silicone tetrachloride, etc. A particularly suitable silica is a product marketed by Godfrey L. Cabot, Inc. as "Cab-O-Sil H5," a pyrogenic silica prepared by the high temperature hydrolysis of silicone tetrachloride.

PREFERRED EMBODIMENT

The following examples are illustrative of the preferred embodiment of the present invention.

EXAMPLE I

A finely divided silica, "Cab-O-Sil H5," was treated with a toluene solution of an amount of hexamethyldisilazane in excess of that calculated to completely cover the surface of the silica. The coating technique used was adapted from the technique used for coating chromatography supports as described in Technical Bulletin No. 12, dated Mar. 1, 1966, issued by Applied Sciene Laboratories, Inc.

A grease was prepared by thickening a silicone polymer oil (a phenylmethyl silicone polymer fluid marketed as "QF6–7024" silicone fluid by Dow Corning) with 13% of the above surface-treated silica.

For the purpose of comparison the same finely divided silica was treated with n-butyl (C_4), isooctyl (iC_8), dodecyl (C_{12}) and cetyl (C_{16}) alcohols to form the corre-

sponding estersils, and greases were prepared by thickening "QF6–7024" silicone fluid with such estersils.

The above greases were subjected to a bearing performance test at 600° F., run at 10,000 r.p.m. in the atmosphere, using a 25 lb. axial and 50 lb. radial load. The test is continued to bearing failure.

The results of this test are tabulated in the following table:

TABLE I

Type of thickener	Percent thickener	Time to failure, hr.
(1) Cab-O-Sil—hexamethyldisilazane	13.0	126
(2) Cab-O-Sil—"Estersil PLI 71" [1]	7.5	37
(3) Cab-O-Sil—iC_8alcohol	14.0	2.5
(4) Cab-O-Sil—C_{12} alcohol	11.1	24
(5) Cab-O-Sil—C_{16} alcohol	13.6	17
(6) Cab-O-Sil—untreated	4.3	40

[1] n-Butanol coated silica marketed by Du Pont.

The above data demonstrate the superior thermal stability of greases thickened with silica-treated hexamethyl disilazones.

Example II.—A C_{11} perfluoroalcohol

$$[H(CF_2—CF_2)_5CH_2OH]$$

was reacted at room temperature with dimethyldichlorosilane in equimolar amounts, to give a fluorosilane, perfluoroalcoxy-dimethyl-chlorosilane.

"Cab-O-Sil H5" was treated for two hours with the above fluorosilane in a reflux system in toluene suspension at the toluene boiling point temperature under a slow stream of nitrogen. Two percent boric acid, based on the silica was added to the mixture of silica and fluorosilane. The relative amounts used were such that one-half of the estimated surface hydroxyls of silica should be covered. Toluene was then distilled off and the thickener dried at 400° F. under slight vacuum.

The following greases were prepared with the above fluorosilane surface-treated silica.

	Percent thickener	Vehicle fluid
Grease A	20	Silicone polymer.[1]
Grease B	15.4	Perfluoro polymer.[2]
Grease C	8.0	Do.

[1] QF6-7024, marketed by Dow Corning.
[2] PR 143 a perfluoro-polymeric fluid prepared by Du Pont.

Greases A and B were subjected to static aging tests at 400° F. and at 600° F. for 18 hours. The results are shown in Table II.

TABLE II

Grease	Aging, 18 hours	
	400° F.	600° F.
A	Stable	Liquid.
B	do	Stable.

The 400° F. and 600° F. aged Grease B samples were combined and additional PR143 fluid added to give a grease containing 6.2% of the thickener. This grease product was stable after aging at 600° F. for an additional 140 hours, for a total aging period of 158 hours.

Grease C, after being subjected to a 450° F. static aging test for 24 hours, was subjected to two high speed bearing performance tests, at 10,000 r.p.m. at 50 lb. radial and 25 lb. axial loading, one at 450° F. and the other at 550° F. At 450° F. the bearing was still running at 1250 hours of operation; at 550° F. the bearing failed after operating 270 hours.

The lubricant grease compositions of the present invention can contain, if desired, lubricant additives well known to the art without departing from the scope of the invention. For example, such greases may contain a corrosion and/or rust inhibitor, an E.P. agent, an antioxidant, a metal deactivator, a stabilizer, an anti-wear agent, and the like. The use of such additives and the amounts thereof depend upon the severty of the conditions to which the grease may be subjected.

Percentages given herein are weight percentages unless otherwise stated.

I claim:

1. The grease composition consisting essentially of a major amount of a normally liquid lubricant and an amount, sufficient to thicken said liquid lubricant to grease consistency, of finely divided silica having the surface thereof treated with the reaction product of equimolar amounts of dimethyldichlorosilane and a perfluoroalcohol having the formula $H(CF_2CF_2)_xCH_2OH$ wherein x is 1 to 8.

2. The composition claim 1 wherein the amount of silica is about 2–40%.

3. The composition of claim 2 wherein said liquid lubricant is a silicone polymer oil.

4. The composition of claim 2 wherein said liquid lubricant is a perfluoro polymer.

5. The composition of claim 4 wherein x is 5 in the perfluoroalcohol formula.

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DANIEL E. WYMAN, Primary Examiner

I. VAUGHN, Assistant Examiner

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