Patented May 20, 1952 2,597,018

UNITED STATES PATENT OFFICE

2,597,018

LUBRICANT

Robert L. Merker, Washington, D. C., and Curtis R. Singleterry, Arlington, Va.

No Drawing. Application May 18, 1948, Serial No. 27,814

8 Claims. (Cl. 252—42.7)

(Granted under the act of March 3, 1883, as amended April 30, 1928; 370 O. G. 757)

1

This invention relates to thickened lubricating compositions such as greases and heavy lubricating fluids.

Lubricating greases and thickened oils have in the past comprised a lubricating oil and a gel-forming agent dispersed therein. The gel-forming agent has been by universal usage a metal soap.

Greases of the prior art have included as thickeners a very wide variety of the metal soaps of the fatty acid series. For normal operating conditions, such compounds have usually been satisfactory. Nevertheless, serious problems have been recognized in the art, and continued effort exerted to provide a solution therefor. These attempts have not in the past been marked with great success.

Among the more serious defects of presently conventional greases, are an insufficient stability in respect to atmospheric oxidation, bleeding, and shear breakdown.

With respect to oxidation, it has been appreciated that a bearing packed with a conventional grease will after a period of time actually become locked as a result of the degradation of the grease. Similar processes take place in greatly shortened periods of time under high temperature application. This is believed to be due to the presence of the metal soap, which acts as an oxidation catalyst for the lubricating oil, particularly at high temperatures.

For high temperature use, conventional greases fail completely because of the relatively low melting point of the soap employed. For many uses, therefore, such as in rolling mills and in combustion turbine engines, conventional greases cannot be employed with any degree of satisfaction.

It is particularly to these problems, as well as others existing in the prior art, that the present invention is directed, and among its objects is the overcoming of the same.

A primary object of the present invention is to provide a method for lubricating a bearing, particularly at high temperatures, and over long periods of storage time. A further object of the present invention is to provide a novel thickened lubricating composition, particularly applicable to high temperature uses and for long periods of storage, but also applicable to all conventional and less exacting uses, and methods of formulating the same.

Other objects and purposes of the present invention will appear hereafter.

As has been noted, basic problems arising in the use of presently conventional greases are due

2

to the employment of a metallic soap as the thickener therein. Not only does the metallic soap act as an oxidation catalyst at elevated temperatures, but also the soap itself melts at relatively low temperatures and undergoes undesirable phase changes at still lower temperatures. Consequently this component of presently used greases renders them of little utility in high temperature applications such as those arising in rolling mills and in combustion turbine engines.

In lubricating compositions of the present invention, however, this factor is not present. The high temperature limitations are imposed by the characteristics of the lubricating oil employed rather than those of the thickening agent. Further, the thickening agent does not act at high temperatures or otherwise as a catalyst for the oxidation of lubricating agent. Consequently lubricating compositions of the present invention are applicable both to high temperature uses and in the lubrication of machinery which may be stored over long periods of time and put into operation without cleaning and relubricating the bearing surfaces thereof. At the same time, the compositions of the present invention are applicable and of high utility in all the more common-place applications of conventional soap base greases.

Conventional greases consist essentially of a gel comprising a lubricating oil and colloidal, dispersed, soap crystals or fibers. The physical structure of the lubricating compositions of the present invention is similar to that of the prior art using soap as the gel-forming agent.

As the lubricating oil phase, non-drying oils may be employed, such as petroleum, diester, silicone, polyethylene and Ucon oils.

As the gel-forming phase of the lubricating composition we employ a phthalocyanine compound.

These materials are characterized by being stable at amazingly high temperatures. Many can be sublimed without decomposition at temperatures in the neighborhood of 500° C. They may be obtained in a physical form suitable for use in lubricating applications. They are compatible with lubricating oils and can be dispersed therein with little difficulty to form a thickened lubricant or grease.

The phthalocyanines are relatively unreactive and are compatible not only with the usual special purpose additives employed in lubricating compositions such as anti-wear, anti-rust, anti-oxidant, and extreme pressure additives, but also are compatible with conventional metal-

lic soap thickeners, if a composite thickened lubricant should be considered desirable. As will be appreciated, the phthalocyanines have been widely used in pigments. Although the color of these pigment compositions is incidental to the purposes of the present invention, it does afford an outstanding characteristic for the identification of these lubricants. In comparison to the catalysis of oxidation by the metallic soaps, it is interesting to note that copper phthalocyanine affords a satisfactory rubber pigment, although it is well known that small traces of ionizable copper act as an oxidation catalyst for rubber.

Compositions of the present invention include not only metal-free phthalocyanine, but metal phthalocyanines, such as zinc, nickel, aluminum and particularly copper phthalocyanine. Chlorinated phthalocyanine and chlorinated metal phthalocyanines may also be employed.

The amount of the phthalocyanine employed is not critical, and may vary from 10% to 45% by weight of the grease mixture. Greater proportions of the phthalocyanine thickener are rarely necessary. Lesser proportions of the phthalocyanine in grease compositions wil result in excessive bleeding if employed alone, but may be used in combination with other thickening agents, such as metallic soaps. However, lesser proportions of phthalocyanine may be used in thickened oil compositions.

The particle size of the phthalocyanine pigments employed is also not critical, and extends over most of the range employed in conventional metallic soap greases. The phthalocyanine crystals may be a tenth of a micron or less in dimensions, but crystals longer than 15 microns are preferably not used in these lubricating compositions.

Compounding lubricants of the present invention differs in many respects from those of conventional metallic soap base greases. In the latter, the components are either heated until the soap liquifies and is then crystalized out in the lubricating oil phase to form the desired crystal dispersion, or a fat is saponified with a metal oxide or hydroxide in the presence of the lubricating oil.

A variety of procedures may be adapted to disperse the phthalocyanine crystals in the lubricating agent. For instance, it has been found that phthalocyanines may be dispersed in lubricating fluids with the aid of such mechanical devices as ball mills and colloid mills. A good dispersing agent for the phthalocyanine, which is soluble in the lubricating agent, may be introduced into the latter and the phthalocyanine pigment then introduced and mixed therewith by mechanical agitation. The dispersing agent may then be removed by evaporation. Benzene is quite satisfactory for this mode of operation.

It is also possible to employ the well known flushing technique wherein the thickener is originally dispersed in water and is subsequently transferred from the water phase to the oil phase by agitating the two phases in contact with each other.

Comparative tests have been run on a series of greases employing various lubricating fluids in combination with copper phthalocyanine and these same fluids in lithium stearate greases of conventional practice. The lubricants included silicones, diesters, Ucon fluids, polyethylenes, and petroleum oils. Oxidation tests in a convection type oven at 200° C. showed that the copper phthalocyanine greases are much more heat and oxidation stable than the corresponding soap type greases containing the same fluid bases. Loaded bearing tests at 160° C. ran for 450 hrs. with a grease composed of copper phthalocyanine and ditetradecyl sebacate, whereas the best di-tetradecyl sebacate lithium stearate grease failed under the same test conditions in about 150 hrs.

The lubricating oils employed may be of a wide variety. For employment in conventional applications petroleum oils may be used. The wide variety of these oils available, and their advantages for particular applications, are well known to those learned in the art. The following examples, without limitation thereto, may be taken as illustrative:

Lubricants	Composition by Weight	ASTM Penetration
	Per cent	
1. Copper phthalocyanine	25	360
Petroleum (Pennsylvania)	73	
Di-lauryl selenide	2	
2. Copper phthalocyanine	25	352
Petroleum (Mid-continent)	73	
Di-lauryl selenide	2	

Non-drying animal and vegetable oils also afford a lubricating component entirely satisfactory for the comprehensive lubricants of the present invention. It will be understood, of course, that drying oils such as tung oil or linseed oil or similar synthetic compositions used in paints are not suitable for lubricating purposes. The following are illustrative:

Lubricants	Composition by Weight	ASTM Penetration
	Per cent	
1. Copper phthalocyanine	20	370
Castor oil	80	
2. Copper phthalocyanine	25	343
Lard oil	80	

Synthetic diester base lubricants may also be employed. Exemplary lubricating compositions of this class are the following:

Lubricants	Composition by Weight	ASTM Penetration
	Per cent	
1. Copper phthalocyanine	25	354
Di-(2-ethylhexyl) sebacate	72	
Di-Lauryl selenide	3	
2. Copper phthalocyanine	25	333
Di-(tetradecyl) sebacate	74	
Phenothiazine	1	
3. Phthalocyanine (metal free)	25	400
Di-(2-ethylhexyl) sebacate	72	
Di-lauryl selenide	3	
4. Chlorinated copper phthalocyanine	44	310
Di-(2-ethylhexyl) sebacate	55	
Phenothiazine	1	

Particularly for high temperature applications silicone fluids may be used. Of these the following are illustrative:

Lubricants	Composition by Weight	ASTM Penetration
	Per cent	
1. Copper phthalocyanine	25	313
Polymethylphenylsiloxane (DC-550)	75	
2. Copper phthalocyanine	20	356
Polymethylsiloxane (DC-500) etc	80	

Other synthetic lubricating compositions may be employed. For instance all of the following compositions may be used: (Ucons).

Lubricants	Composition by Weight	ASTM Penetration
	Per cent	
1. Copper phthalocyanine	25	
Polyalkylene oxide (Ucon L B-400 x)	74	344
Phenothiazine	1	
2. Copper phthalocyanine	20	
Polyalkylene oxide (Ucon L B-400 x)	79	333
Phenothiazine, etc.	1	

In view of the unreactive nature of the gel forming agents of the present invention other lubricating fluids may be employed therewith to form thickened lubricants.

A representative grease of the present invention would include the following as essential components of the present invention:

Lubricating oil 55% to 85%
Phthalocyanine 10% to 45%

Lower percentages of the phthalocyanine may be employed where it is used in combination with another thickener.

A thickened oil of the present invention would include the following essential components of the present invention:

Lubricating oil 90%–99.9%
Phthalocyanine 0.1%–10%

The lubricating compositions of the present invention can be introduced to bearing surfaces by the mechanical expedients usually employed for other lubricants, as for instance, wicks, rings, pressure lines, or by packing a bearing provided with a retainer.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

What is claimed is:

1. A thickened lubricating composition comprising a lubricating oil and up to about 45% by weight of a phthalocyanine dispersed therein in a proportion sufficient to cause gel formation in the oil.

2. The composition of claim 1 wherein the lubricating oil comprises essentially a petroleum lubricant.

3. The composition of claim 1 wherein the lubricating oil comprises essentially a silicone lubricant.

4. The composition of claim 1 wherein the lubricating oil comprises essentially a synthetic diester base lubricant.

5. A lubricating grease comprising a lubricating oil and from 10% to 45% by weight the phthalocyanine thickener.

6. A thickened lubricating composition comprising a lubricating oil and up to about 45% by weight of a metal phthalocyanine dispersed therein in a proportion sufficient to cause gel formation in the oil.

7. A thickened lubricating composition comprising a lubricating oil and up to about 45% by weight of copper phthalocyanine dispersed therein in a proportion sufficient to cause gel formation in the oil.

8. A thickened lubricating composition comprising a lubricating oil and up to about 45% by weight of chlorinated copper phthalocyanine dispersed therein in a proportion sufficient to cause gel formation in the oil.

ROBERT L. MERKER.
CURTIS R. SINGLETERRY.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,659,149	Nill	Feb. 14, 1928
1,937,463	Nill	Nov. 28, 1933
1,996,391	Straus	Apr. 2, 1935
2,361,009	Carman et al.	Oct. 24, 1944
2,388,887	Weissberger et al.	Nov. 13, 1945
2,390,943	Kavanagh et al.	Dec. 11, 1945
2,476,952	Beard	July 26, 1949

FOREIGN PATENTS

Number	Country	Date
517,475	Great Britain	Jan. 31, 1940