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LUBRICANT

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This invention relates to lubricants and more particularly to filled lubricants having extreme pressure characteristics prepared by the addition to ordinary lubricating oils and greases of solid fillers as hereinafter disclosed, which fillers have the effect of minimizing friction and reducing bearing wear even under heavy duty conditions.

The advantage accruing from the use of certain solid type additives in lubricating oils and greases has long been recognized in the art. The literature contains references to a wide variety of fillers which may be classified according to their apparent function as follows:

1. Those materials which are in themselves lubricants in the dry state and merely enhance the lubricating qualities of the grease, or are intended to remain after the grease has been volatilized by the temperature of operation. Into this class would be placed for example graphite and mica, both of which materials have been known and used for many years.

2. Those materials which fill unevenness in the rough bearing and thus lower friction by exposing a smoother, padded bearing surface. This class seems most applicable to large, slow-speed, low-pressure bearings where abrasion is very apt to be a serious problem and in this category would be placed such fillers as asbestos fiber, wood flour, wool yarn, fish scales, charcoal, and the like.

3. Those materials which exert a polishing action and find application by virtue of their ability to abrade the surfaces yielding as a result a smoother bearing. Such fillers seem to be of more value in high-speed, low-load bearings where high frictional heat, due to rough bearings is a predominate factor. This class appears definitely detrimental when a heavy load with resultant extreme pressures is applied. Under such conditions of high load abrasion of the bearing becomes excessive and it very rapidly disappears. Materials showing this abrading and polishing action, for example, are emery dust, zinc oxide, alum, red lead, iron filings, and the like.

4. The fourth class appears to hold the greatest promise for advance in the field of fillers, and especially in the field of fillers to be employed in extreme pressure lubricants. Just as in gear oils and the like where a load-carrying additive is required, so there appears to be a need for such an additive for greases. Only a few materials have so far been disclosed in the literature which may properly be grouped in this class. However, recent patents have shown the calcium, magnesium and zinc salts of the phosphates, silicates

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and borates to exert the necessary surface action of such a nature that they may be rightly classified in this group 4.

It should be pointed out that distinction between classes 3 and 4 is extremely difficult without actual test, as materials which might be assumed to be abrasive are found to exhibit unusual surface active properties. In such cases particle size would of course be of extreme importance.

It is an object of my invention to provide a novel class of solid materials which may be added to lubricating oils and greases to give improved filled lubricants having extreme pressure properties.

It is a more particular object of my invention to provide a class of fillers for lubricating greases falling in the fourth class of the above categories exerting a beneficial effect because of surface active properties.

It is another object of my invention to prepare a lubricant which by the addition thereto of fillers as hereinafter disclosed has improved extreme pressure characteristics.

More specifically it is an object of my invention to prepare a lubricant having improved extreme pressure characteristics by the addition thereto of minor amounts of the fillers according to this invention which fillers do not cause the breakdown of the grease structure in operation and which do not function as abrasives towards the bearings in which they come in contact.

Other objects and advantages of my invention will become apparent to those skilled in the art as the description thereof proceeds.

I have found that the incorporation of a small portion of a finely powdered, non-crystalline metallic sulfide materially enhances the bearing-wear characteristics and the load-carrying capacity of a normal grease.

More specifically I have found that the higher sulfides, providing they are non-crystalline, of metals capable of uniting with sulfur in more than one ratio are highly active in preventing failure of a heavily loaded bearing. Included in this classification are such metallic sulfides as antimony pentasulfide, arsenic pentasulfide, barium trisulfide, ferric sulfide, selenium sulfide, sodium monosulfide, sodium pentasulfide, chromium sulfide, mercuric sulfide, molybdenum tetrasulfide, and the like. The non-crystalline sulfides of the polyvalent metals are the preferred class of compounds and more particularly antimony pentasulfide, mercuric sulfide and stannic sulfide. I have also found that certain metallic

sulfides such as for example zinc sulfide, and mercurous sulfide may be prepared in either crystalline or amorphous form and in the latter instance are desirable as fillers for lubricants according to my invention.

On the basis of numerous tests I have found certain restrictions and conditions which must be taken into consideration in the use of these sulfide type fillers. In addition to the stipulation that they must be non-crystalline and that they preferably are the higher sulfides of metals capable of forming compounds with sulfur in more than one ratio, the sulfide must be one which is stable at normal atmospheric conditions of temperature and of humidity and of such physical state that it may be sufficiently finely powdered so that the abrasive action, if any, will not exceed the action as a surface film producing material. One further restriction in the use of this type of additive is that the grease should preferably be one with a high melting point. It must also be remembered that fillers have limitations in regard to applications for which they are suitable. Filled greases for example have several types of functions other than bearing lubrication such as thread compounds, cable and gear lubricants, and the like.

It is thought that the activity of the particular sulfides as disclosed above is due to the thermal and pressure decomposition of the sulfide to liberate elemental sulfur in a highly active form which functions to provide a sulfide bearing surface as in a typical sulfurized extreme pressure lubricant. However, I do not wish to limit my invention to any theory as to the mechanics of their operation.

The testing of the filled greases presents a variety of problems due primarily to the wide range of functions performed by the various fillers. The following test has been found very useful in segregating and classifying fillers of class 4 above. A modification of the Almen test was used in which three instead of the usual two bushings were employed. A description of the Almen test apparatus and method of operation is included in a paper entitled "Extreme Pressure Lubricants" presented by Wolf and Mougey at the thirteenth annual meeting of the American Petroleum Institute at Houston, Texas, on November 17, 1932. In my modification of this test method two bushings are located on the under side of the test pin, one at each end, and the third or load bushing is located on top, straddling the gap formed by the lower bushings. Whereas, the cup on the Almen test is designed for use with a liquid, it was felt that due to the structure of the greases to be tested an unfair comparison would be obtained because of the failure of the grease to feed properly between the bushings if they were arranged according to the standard method. By use of three bushings, as described above, all parts of the test pin are in contact with a portion of the grease for at least one-half of its circumference thus insuring sufficient contact to provide maximum lubrication. Another modification of the standard test has been to apply a constant load for a variable time. To date all tests have been made by allowing the bearing to run 30 seconds without load and subsequently loading the arm at the rate of 2 pounds per 10 seconds until an arm load of 8 pounds is reached; and then allowing the test to run either until failure occurs or for a definite period of time, such as for example two hours. In this apparatus each two pound weight is

equivalent to 1,000 pounds per square inch projected bearing area thus a total of 4,000 pounds per square inch is obtained. These conditions for operating the machine appear to give the best correlation with actual service data on the performance of extreme pressure lubricants.

In general fillers of classes 1 and 2 above fail before the full load is applied or shortly thereafter, with seizure and shearing of the test pin. Class 3 fillers run for a somewhat greater length of time giving a uniform abrasion and polishing of the test piece until the amount of abraded metal particles becomes great enough to cause scoring with subsequent seizure and failure. Almost all pins used in tests with filled greases using a filler of class 4 have shown a highly uniform polish at the end of the test, although the lapse of time before seizure varies over a comparatively wide range.

In one series of tests to determine the relative effectiveness of the various fillers a standard light grade of barium soap grease was employed having the following composition:

Composition

	Barium soap	Weight, Per Cent	25.00
25	Free alkali	do	0.02
	Free fat	do	0.30
	Water	do	0.20
	Glycerin	do	0.23
30	Dye	do	0.005
	Mineral oil	do	74.245
	S. U. Vis. at 100° F., secs		440
	S. U. Vis. at 130° F., secs		185
	S. U. Vis. at 210° F., secs		54
35	Viscosity index		50
	Flash, COC, °F		410
	Pour point, °F		90

The characteristics of this grease are:

Tests

40	A. S. T. M. unworked pen./77° F	345
	A. S. T. M. worked pen./77° F	335
	A. S. T. M. dropping point, °F	400+
45	Sulfated ash, percent	12.5

Employing this grease, numerous tests were made by the above procedure as to the effectiveness of various metallic sulfide type fillers together with other fillers for comparative purposes. The results of these tests are given in Table I below:

	Filler	Pin	Time, Min.	Comments
55	None	Steel	60	Scored and seized.
	5% Sb ₂ S ₃	do	120	Pin darkened.
	1% Sb ₂ S ₃	do	120	Pin darkened—grease darkened adjacent to pin.
	5% Aluminum hydrate	do	16	Severely scored previous to seizure; sheared.
60	5% PbO	do	15	Severely scored previous to seizure; sheared.
	3% Sb ₂ S ₃	Brass	120	Pin very black. No sign of scoring.
	5% HgS	Steel	70	Slight darkening—highly polished—slightly scored.
65	5% SnS ₂	do	120	Pin black—highly polished—very good.

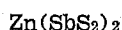
It is clearly evident from this table that the sulfides and particularly antimony sulfide materially improve the extreme pressure characteristic of this grease. A whole series of tests have been made employing various base greases in which I have found that antimony pentasulfide is superior to the other metallic sulfides tested followed by stannic sulfide and mercuric sulfide, thus substantiating results given in the above

table. I do not wish to limit myself, however, to these three metallic sulfides but only point out a consistency of the comparison obtained using various base greases.

These fillers may be incorporated in the desired greases in a number of ways. In some cases for example it may be desirable to saturate the filler with a mineral oil, a fatty oil or a mixture of the two to aid in blending with the grease. However, the addition of the finely powdered metallic sulfides as hereinbefore disclosed may be made directly to the grease with means provided for intimately mixing and dispersing said fillers throughout the body of the grease. Lumpiness or local concentrations of the sulfide should of course be avoided. Methods of incorporating such fillers in greases are well known in the art and are not contemplated by my invention.

My invention is therefore carried into effect by very thoroughly mixing or dispersing in any desirable manner from about one to about ten percent and preferably from about two to about six per cent of a finely powdered, non-crystalline metallic sulfide such as antimony pentasulfide in an oil or grease base possessing in itself good lubricating qualities. For this purpose any desirable oil or grease may be employed, however, I have shown that the high melting point non-hydrous greases are superior for this purpose to the lower grades of grease.

I do not wish to limit my invention to the use of fillers selected solely from the simple non-crystalline metallic sulfides inasmuch as I have found that thiosalts or complex metal sulfides are excellent fillers for extreme pressure lubricants. Complex zinc-antimony sulfides, such as



zinc-tin sulfides and related compounds wherein the metals within the complex may be at different oxidation levels may be employed.

Whereas, the non-crystalline metal sulfides as hereinbefore disclosed may be advantageously employed in lubricating oils as well as greases to impart extreme pressure properties thereto they appear to be particularly effective in grease structures and such usage constitutes the preferred embodiment of my invention.

While the preferred extreme pressure additives of the invention have been described, I do not intend to be limited to the specific compounds mentioned for it is apparent from the foregoing description and examples that a new class of fillers for lubricants and particularly for imparting extreme pressure characteristics to said lubricants has been discovered and many variations and modifications in the utilization of these ma-

terials may occur to those skilled in the art without departing from the spirit and scope of the following claims.

I claim:

1. A composition of matter comprising a lubricant of the class consisting of oils and greases and a small amount, sufficient to impart extreme pressure properties to said lubricant, of a finely divided, non-crystalline metallic sulfide dispersed therein.
2. A composition of matter as in claim 1 wherein the finely divided non-crystalline metallic sulfide is selected from the sulfides of the polyvalent metals.
3. A composition of matter comprising a lubricant of the class consisting of oils and greases and from about 1% to about 10% of a finely divided, non-crystalline metallic sulfide dispersed therein.
4. A composition of matter comprising a lubricant of the class consisting of oils and greases and from about 1% to about 10% of finely divided antimony pentasulfide dispersed therein.
5. A composition of matter comprising a lubricant of the class consisting of oils and greases and from about 1% to about 10% of finely divided mercuric sulfide.
6. An extreme pressure lubricant of the class consisting of oils and greases to which has been added from about 1% to about 10% of finely divided stannic sulfide.

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