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(54) **Grease composition and additive for improving bearing life**

(57) Grease compositions, methods for using the grease compositions, additives for grease compositions, and equipment and vehicles containing the grease compositions for improved fretting wear performance. The grease composition includes a fully formulated grease base and an effective amount of a fretting wear reducing

additive. The fretting wear reducing additive is a metal-free, oil soluble phosphorous-containing, sulfurized amine salt which is used in an amount sufficient to provide a fretting wear weight loss of less than about 20 milligrams.

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Description**TECHNICAL FIELD:**

5 **[0001]** The disclosure relates to a lubricating grease composition for rolling and rotating bearings, use thereof, and more particularly to an improved lubricating grease composition that provides improvements in resistance to fretting developed by repeated impact and oscillation caused by exterior oscillation as occurring during transportation and dropping impact developed by dropping during transportation. The compositions are particularly useful for bearings having inadequate lubrication as a result of non-use.

BACKGROUND AND SUMMARY:

10 **[0002]** Fretting wear (also known as "fretting corrosion" or "false brinelling") is caused by vibration of bearings as is experienced by wheel bearings in cars transported by rail car or roller bearings for industrial equipment stored in areas that experience vibrations from other operating equipment. Fretting wear can cause premature failure of the bearings shortly after start up of the vehicles or equipment. This type of wear phenomena is caused by a short oscillating arc of the bearings which does not allow adequate lubricant flow to the bearing region and traps wear debris adjacent to the bearings. Corrosive oxidation may also take place at the surface of the bearings which may exacerbate wear and damage to the bearings. All of these conditions in turn increase the severity of bearing wear.

15 **[0003]** Fretting wear is primarily experienced in bearings that are not operational for periods of time since regular operation of the bearings will tend to distribute protective lubricant to the bearing area and flush wear debris away from the bearing area. Fretting wear is determined by an industry standard test ASTM D-4170-97(2002) entitled "Standard Test Method for Fretting Wear Protection by Lubricating Greases" which measures the amount of weight loss of a test piece after 20 hours under oscillating conditions. Several key specifications in the grease industry require low values of fretting wear as determined by the foregoing ASTM D-4170 test. Such specifications include, but are not limited to, NLGI's GC-LB specification (<10 mg fretting wear), GM's LS2 specification for "High-Temperature", "High-Speed Bearing", and "Multipurpose" greases (<10 mg fretting wear), and GM's LS2 specification for "Fretting and Corrosion Inhibiting" greases (<2 mg fretting wear). Accordingly, a continual need exists for improved grease formulations that meet industry specifications for fretting wear without detrimentally affecting other important properties of the greases.

20 **[0004]** In view of the continuing need for improved grease compositions, exemplary embodiments of the disclosure provide grease compositions, additives for grease compositions, methods for using the grease compositions, and equipment and vehicles containing the grease compositions for improved fretting wear performance. The grease composition includes a fully formulated grease base and an effective amount of fretting wear reducing additive. The fretting wear reducing additive is a metal-free, oil soluble phosphorous-containing, sulfurized amine salt that is used in an amount sufficient to provide a fretting wear weight loss of less than about 20 milligrams.

25 **[0005]** Another exemplary embodiment provides a method for reducing fretting wear in a bearing. According to the method, a grease composition is applied to the bearing. The grease composition includes a fully formulated grease base and an effective amount of additive comprising a metal-free, oil soluble phosphorous-containing, sulfurized amine salt sufficient to provide a fretting wear weight loss of less than about 20 milligrams.

30 **[0006]** Yet another exemplary embodiment of the disclosure provides a vehicle wheel bearing in contact with a fully formulated grease base and an effective amount of additive comprising a metal-free, oil soluble phosphorous-containing, sulfurized amine salt sufficient to provide a fretting wear weight loss of less than about 20 milligrams.

35 **[0007]** Another exemplary embodiment of the disclosure provides an additive for a grease which provides a reduction in fretting wear weight loss characteristics of the grease. The additive is a metal-free oil soluble phosphorous-containing, sulfurized amine salt component that when added to the grease in a predetermined amount provides a grease having a fretting wear weight loss of less than about 20 milligrams.

40 **[0008]** The additive for the grease compositions disclosed herein is highly compatible with the grease compositions. Accordingly, exemplary embodiments of the disclosure may provide increased flexibility for formulating greases while providing enhanced fretting wear protection without significantly altering other performance properties (such as extreme pressure properties and corrosion protection) of the grease formulations that may be reduced by competition with surface active components. In some formulations, the additive may also noticeably enhance antiwear, extreme pressure, and corrosion protection properties of the grease formulation.

DETAILED DESCRIPTION OF EXEMPLARY EMBODIMENTS:

45 **[0009]** Grease formulations which may be enhanced by the additive described herein include a wide variety of conventional mineral and synthetic oil-based grease compositions. Such compositions include, but are not limited to, greases containing metal complex thickeners, alkali metal complex thickeners, and urea compound thickeners. In addition to the

additive described herein, the fully formulated grease formulations may include other additives useful for obtaining specific performance properties.

[0010] A wide variety of base oils may be used to prepare the grease compositions described herein. For example, the base oil may be selected from any of the conventionally used mineral oils, synthetic hydrocarbon oils, or synthetic ester oils. In general such oils may have a viscosity in the range of from about 5 to about 10,000 cSt at 40° C., although typical applications may require an oil having a viscosity ranging from about 10 to about 1,000 cSt at 40° C. Mineral oil basestocks which may be used may be any conventionally refined base stocks derived from paraffinic, naphthenic, and mixed base crudes. Synthetic oil basestocks that may be used include esters of dibasic acids such as di-2-ethylhexyl sebacate, esters of glycols such as C₁₃-oxide acid diesters of tetraethylene glycol, or complex esters such as those formed from 1 mole of sebacic acid, 2 moles of tetraethylene glycol, and 2 moles of 2-ethylhexanoic acid. Other synthetic oils that may be used include synthetic hydrocarbons such as polyalphaolefins; alkyl benzenes, alkylate bottoms from the alkylation of benzene with tetrapropylene or with the copolymers of ethylene and propylene; silicon oils, e.g. ethyl phenyl polysiloxanes, methyl polysiloxanes, etc.; polyglycol oils, e.g. those obtained by condensing butyl alcohol with propylene oxide; carbonate esters, e.g. the product of reacting C₈-oxo alcohol with ethyl carbonate to form a half ester followed by reaction of the latter with tetraethylene glycol; and the like. Other suitable synthetic oils include the polyphenyl ethers, e.g. those having from about 3 to about 7 ether linkages and about 4 to about 8 phenyl groups. (See U.S. Pat. No. 3,424,678, column 3.) The amount of base oil in the grease composition may also vary broadly, but, typically, will comprise a major portion, for example from about 75 to about 95 wt. %, of the grease composition.

[0011] Grease compositions as described herein also contain a minor proportion of a simple soap or a complex soap thickening agent or of an organic thickener or inorganic clay thickener. Such grease types include, but are not limited to, alkali metal or alkaline earth metal simple and complex soap greases, metal simple and complex greases, as well as polyurea greases, organo-clay greases and bentonite clay/swelling-clay type thickeners.

[0012] As an example, alkali or alkaline earth metal complex soap greases have found a major use as greases for bearings, particularly sealed bearings (e.g. in automobiles and electrical appliances). Sealed bearing greases must meet numerous performance requirements including extended bearing life, high temperature performance, high dropping point, and defined requirements relating to oil separation, oxidation stability, fretting wear protection and low noise. However, although offering long service and high temperature benefits, such greases often suffer from generally higher noise characteristics than an alkali or alkaline earth metal simple grease.

[0013] For alkali or alkaline earth metal complex soap greases, useful thickeners contain two, typically, three alkali or alkaline earth metal components. The first may be an alkali or alkaline earth metal soap of at least one, hydroxy fatty acid, such as C₁₂ to C₂₉ hydroxy acid. The second component may be selected from a alkali or alkaline earth metal compound of (i) a C₂ to C₁₂ aliphatic or cycloaliphatic dicarboxylic acid (or C₁ to C₁₉, such as C₁ to C₄, alkyl ester thereof); or (ii) of a C₃ to C₂₄ hydroxy carboxylic acid (or C₁ to C₁₉ such as C₁ to C₄, alkyl ester thereof) which has the hydroxy group separated from the carboxyl group by six or less carbon atoms; or a mixture thereof.

[0014] Useful hydroxy fatty acids include hydroxystearic, hydroxy-ricinoleic, hydroxybehenic and hydroxypalmitic. An especially useful hydroxyl fatty acid is hydroxystearic acid. The second alkali or alkaline earth metal compound is suitably a C₆ to C₁₀ aliphatic dicarboxylic acid, more typically azelaic or sebacic acids, especially azelaic acid, or said ester of any of these. The C₃ to C₂₄ hydroxy-carboxylic acid is typically lactic acid, salicylic acid or other hydroxy-benzoic acid, more suitably salicylic acid or an ester of any of the foregoing. The amount of alkali or alkaline earth metal soap complex thickening agent in the grease composition may range from about 5 to about 20 wt. %. The weight ratio of hydroxy fatty acid to aliphatic dicarboxylic acid and/or hydroxy-carboxylic acid is typically from about 10:0.5 to about 10:15, for example from about 10:1.5 to about 10:6.

[0015] Other thickening agents include, but are not limited to, salt and salt-soap complexes such as: calcium stearate-acetate (U.S. Patent No. 2,197,263); barium stearate-acetate (U.S. Patent No. 2,564,561); calcium stearate-caprylate-acetate complexes (U.S. Patent No. 2,999,065); calcium caprylate-acetate (U.S. Patent No. 2,999,066); and calcium salts and soaps of low-(1-6 carbon atoms), intermediate-(7-12 carbon atoms) and high-(13 to 18 carbon atoms) molecular weight acids and of nut oil acids.

[0016] Another group of thickening agents includes substituted ureas, phthalocyanines, indanthrene, pigments including perylimides, pyromellitimides, and ammeline.

[0017] Examples of metal soaps which may be effective thickening agents for grease compositions according to the disclosed embodiments may include aluminum laurate, aluminum soap oleate, aluminum stearate, aluminum benzoate stearate, aluminum benzoate oleate, aluminum benzoate 12-hydroxy stearate, aluminum toluate stearate, aluminum benzoate naphthenate, aluminum benzoate hydrogenated rosin, aluminum benzoate sulfonate, aluminum azelate stearate, aluminum phosphate benzoate stearate, aluminum benzoate hydroxy stearate, lithium laurate, lithium stearate, lithium benzoate stearate, lithium benzoate oleate, lithium benzoate 12-hydroxy stearate, lithium toluate stearate, lithium benzoate naphthenate, lithium benzoate hydrogenated rosin, lithium benzoate sulfonate, lithium azelate stearate, lithium phosphate benzoate stearate, lithium benzoate hydroxy stearate, and the like.

[0018] The metal complex soap or alkali or alkaline earth metal complex may be present in the grease in an amount

sufficient to thicken the oil to the consistency of a grease. Broadly speaking, the amount of soap or alkali metal complex may range from about 1 to about 30 wt. % of the grease. Typically, however, from about 5 to about 20 wt. %, for example from about 10 to about 15 wt. %, of the thickening agent may be used to thicken the grease.

[0019] In addition to the components already mentioned, a fully formulated grease composition may also contain minor amounts of other additives which include, but are not limited to, corrosion inhibitors, antiwear agents, pour point depressants, tackiness agents, extreme pressure agents, viscosity improvers, oxidation inhibitors, rust inhibitors, dyes, and the like. Such components may be present in the fully formulated grease composition in amounts ranging from about 1 to about 10 percent by weight.

[0020] An important component of a grease composition according to the disclosure is an effective amount of a fretting wear reducing agent. A particularly suitable additive for reducing fretting wear is an oil-soluble metal-free salt of a phosphorous-containing compound. The oil-soluble metal-free phosphorus-containing compounds are for the most part partially or fully esterified acids of phosphorus. Such compounds include for example phosphates, phosphites, phosphonates, phosphonites, and their various sulfur analogs. Examples include monohydrocarbyl phosphites; monohydrocarbyl phosphates; monohydrocarbyl mono-, di-, tri-, and tetrathiophosphites; monohydrocarbyl mono-, di-, tri-, and tetrathiophosphates; dihydrocarbyl phosphites; dihydrocarbyl phosphates; dihydrocarbyl mono-, di-, tri-, and tetrathiophosphites; dihydrocarbyl mono-, di-, tri-, and tetrathiophosphates; trihydrocarbyl phosphites; trihydrocarbyl phosphates; trihydrocarbyl mono-, di-, tri-, and tetrathiophosphites; trihydrocarbyl mono-, di-, tri-, and tetrathiophosphates; the various hydrocarbyl phosphonates and thiophosphonates; the various hydrocarbyl phosphonites and thiophosphonites, and analogous oil-soluble derivatives of polyphosphoric and polythiophosphoric acids; and many others. A few specific examples of such compounds are tricresyl phosphate, tributyl phosphite, triphenyl phosphite, tri-(2-ethylhexyl) phosphate, dihexyl thiophosphite, diisooctyl butylphosphonate, tricyclohexyl phosphate, cresyl diphenyl phosphate, tris (2-butoxyethyl) phosphite, diisopropyl dithiophosphate, tris(tridecyl)tetrathiophosphate, tris(2-chloroethyl) phosphate, and like compounds.

[0021] Particularly suitable metal-free phosphorus-containing compounds for use herein are (a) the oil-soluble amine salts of monohydrocarbyl monothiophosphoric acids, (b) the oil-soluble amine salts of dihydrocarbyl monothiophosphoric acids, and (c) combinations of (a) and (b). Such compounds may be made by reacting a mono- and/or dihydrocarbyl phosphite with sulfur or an active sulfur-containing compound such as, sulfur, dihydrocarbyl polysulfides, sulfurized olefins, sulfurized fatty acid esters, trithiones, sulfurized thienyl derivatives, sulfurized terpenes, sulfurized oligomers of C₂-C₈ monoolefins, and sulfurized Diels-Alder adducts, and one or more primary or secondary amines. Such reactions tend to be highly exothermic reactions which may become uncontrollable, if not conducted properly. A suitable method of forming amine salts as described above involves a process which includes (i) introducing, at a rate such that the temperature does not exceed about 60° C., one or more dihydrocarbyl hydrogen phosphites, such as a dialkyl hydrogen phosphite, into an excess quantity of one or more active-sulfur-containing materials, such as sulfurized branched-chain olefin (e.g., isobutylene, diisobutylene, triisobutylene, etc.), while agitating the mixture so formed, (ii) introducing into this mixture, at a rate such that the temperature does not exceed about 60° C., one or more aliphatic primary or secondary amines, for example one or more aliphatic primary monoamines having in the range of about 8 to about 24 carbon atoms per molecule while agitating the mixture so formed, and (iii) maintaining the temperature of the resultant agitated reaction mixture at between about 55° and about 60° C. until the reaction is substantially complete. Another suitable way of producing these amine salts is to concurrently introduce all three of the reactants into the reaction zone at suitable rates and under temperature control such that the temperature does not exceed about 60° C.

[0022] The amount of fretting wear reducing agent in a fully formulated grease composition may range from about 0.1 to about 10 percent by weight. Useful amounts of the fretting wear reducing agent range from about 0.5 to about 5 percent by weight based on the total weight of the grease composition.

[0023] In order to illustrate aspects of the disclosed embodiments, the following nonlimiting examples are provided. In the examples, the fretting wear reducing agent was added to two commercially available grease compositions. The first grease composition contained a major amount of paraffin base oil and a minor amount of aluminum benzoate stearate complex thickening agent, antimony dithiocarbamate, molybdenum disulfide, and/or tackiness agents, and rust/oxidation inhibitors, hereinafter referred to as "Aluminum Complex Grease." The second grease composition contained a major amount of base oil, and a minor amount of lithium hydroxyl stearate complex, zinc dialkyl dithiophosphate, and diphenylamine, hereinafter referred to as a "Lithium Complex Grease (1)." The third grease composition included about 4.5 wt.% of an additive package containing zinc dialkyldithiophosphate, sulfurized isobutylene, a corrosion inhibitor, and several antioxidants, hereinafter referred to as a "Lithium Complex Grease (2)." The fretting wear reducing agent was an oil-soluble mixed amine salt of dihydrocarbyl monothiophosphoric acid made by sulfurization of dibutyl hydrogen phosphite in the presence of oleyl amine and n-octylamine as generally described in U.S. Patent No. 5,328,619. In each of the runs, the fretting wear weight loss was determined using an industry standard ASTM D-4170-97(2002) that measures weight loss of the test piece after 20 hours under oscillating conditions.

Example 1**[0024]****Table 1**

Run No.	Fretting Wear Reducing Agent Wt. %	Aluminum Complex Grease Wt. Loss (mg)	Lithium Complex Grease (1) Wt. Loss (mg)	Lithium Complex Grease (2) Wt. Loss (mg)
1	0.0	22.4	9.3	39.9
2	0.0	30	---	---
3	0.5	19.5	8.6	10.9
4	1.0	14.3	---	---
5	1.0	17.8	8.4	---
6	1.5	11.1	---	---
7	2.0	4.2	7.2	---

[0025] As shown by Runs 3-5 in the above table, an Aluminum Complex Grease containing from 0.5 to 2.0 wt.% of the additive showed a dramatic reduction in fretting wear (4.2 to 19.5 mg weight loss) compared to the same grease without the additive illustrated in Runs 1 and 2 (22.4 to 30 mg weight loss). The Lithium Complex Grease (1) showed a less dramatic drop but was still significant as the fretting wear was lowered from 9.3 mg weight loss to 7.2 mg weight loss with 2 wt.% of the additive. However, Lithium Complex Grease (2) had about a 73% decrease in weight loss when 0.5 wt. % of the fretting wear reducing agent was used compared to Run 1 where none of the fretting wear reducing agent was used.

[0026] The fretting wear reducing agent may also be effective to reduce corrosion and increase extreme pressure (EP) properties of a grease. In the following example, the above grease formulations were subjected to a 4-Ball extreme pressure test to determine the weld point and/or 4-ball wear under load. Also, the Lithium Complex Grease (2) was subjected to a bearing corrosion test. The results of the foregoing tests are provided in the following table.

Example 2**[0027]****Table 2**

Run No.	Fretting Wear Reducing Agent Wt. %	Aluminum Complex Grease 4-Ball EP (kg weld)	Lithium Complex Grease (1) 4-Ball wear (mm)	Lithium Complex Grease (2) 4-Ball EP (kg weld)
1	0.0	230	0.475	300
2	0.0	250	---	---
3	0.5	250	---	310
4	1.0	250	---	---
5	1.0	260	---	---
6	1.5	250	---	---
7	2.0	260	0.402	---

[0028] In the foregoing Table 2, the Aluminum complex grease without the fretting wear reducing agent had weld points of between 230 and 250 kg while the addition of 1 to 2 wt.% of fretting wear reducing agent raised the weld points to 260 kg. Additionally, the fretting wear reducing agent at 0.5 wt.% improved the Lithium Complex Grease (2) from a 300 kg weld point to a weld point of 310 kg. The fretting wear reducing agent also improved the 4-Ball wear scar of the Lithium Complex Grease (1) about 15% over the same grease without the fretting wear reducing agent.

[0029] Additionally, a 48 hour bearing corrosion test at 52° C. was conducted on bearings using the Lithium Complex

Grease (2) with and without the fretting wear reducing agent. Only one out of three bearings had no corrosion when the Lithium Complex Grease (2) contained no fretting wear reducing agent which is considered a failure by the ASTM D-1743 test method. However, a pass condition was obtained with three out of three bearings having no corrosion when the Lithium Complex Grease (2) contained 0.5 wt.% of the fretting wear reducing agent.

[0030] While the foregoing examples showed significant fretting wear reduction in aluminum complex and lithium complex greases, it is believed that the additive is suitable for reducing the fretting wear of any type of grease. Accordingly, a grease containing the additive as described herein may be used to reduce fretting wear in vehicle wheel bearings, in rotating and sliding equipment bearings, in electrical appliances, ball-and-roller bearings used in various types of electronic recording and reading apparatuses such as hard disk drives, video tape recorders, laser beam printers, compact disk drives, digital video disk drives, and other applications where fretting wear may be present due to non-use or vibrations and/or inadequate lubrication of moving parts.

[0031] At numerous places throughout this specification, reference has been made to a number of U.S. Patents and publications. All such cited documents are expressly incorporated in full into this disclosure as if fully set forth herein.

[0032] The foregoing embodiments are susceptible to considerable variation in its practice. Accordingly, the embodiments are not intended to be limited to the specific exemplifications set forth hereinabove. Rather, the foregoing embodiments are within the spirit and scope of the appended claims, including the equivalents thereof available as a matter of law.

[0033] The patentees do not intend to dedicate any disclosed embodiments to the public, and to the extent any disclosed modifications or alterations may not literally fall within the scope of the claims, they are considered to be part hereof under the doctrine of equivalents.

Claims

1. A grease composition comprising a fully formulated grease base and an effective amount of additive comprising a metal-free, oil soluble phosphorous-containing, sulfurized amine salt sufficient to provide a fretting wear weight loss of less than 20 milligrams.
2. The grease composition of claim 1 wherein the fretting wear loss is less than 10 milligrams.
3. The grease composition of claim 1 or 2 wherein the metal-free, oil soluble phosphorous-containing amine salt is derived from a sulfur source and a dihydrocarbyl phosphite.
4. The grease composition of claim 3 wherein the dihydrocarbyl phosphite comprises a dialkyl hydrogen phosphite having from 4 to 25 carbon atoms independently in each alkyl group.
5. The grease composition of claim 3 or 4 wherein the sulfur source is selected from the group consisting of sulfur, dihydrocarbyl polysulfides, sulfurized olefins, sulfurized fatty acid esters, trithiones, sulfurized thienyl derivatives, sulfurized terpenes, sulfurized oligomers of C₂-C₈ monoolefins, and sulfurized Diels-Alder adducts.
6. The grease composition of any one of the preceding claims wherein the amine of the amine salt comprises an aliphatic primary or secondary amine containing from 8 to 24 carbon atoms per molecule.
7. The grease composition of any one of the preceding claims which comprises from 0.1 to 5 wt% of the additive.
8. A wheel bearing comprising the grease composition of any one of the preceding claims
9. The wheel bearing of claim 8 which is a vehicle wheel bearing.
10. A moving part having a bearing surface wherein the bearing surface is lubricated with the grease composition of any one of claims 1 to 7.
11. Equipment containing one or more bearings and comprising the grease composition of any one of claims 1 to 7.
12. Electronic information recording and reading apparatuses containing ball-and-roller bearings and comprising the grease composition of any one of claims 1 to 7.
13. A method for reducing fretting wear in a bearing, comprising applying a grease composition as defined in any one

of claims 1 to 7 to the bearing.

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European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 07 25 1000

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