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(54) **GREASE COMPOSITION**

SCHMIERFETTZUSAMMENSETZUNG

COMPOSITION DE GRAISSE

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- **PATENT ABSTRACTS OF JAPAN vol. 013 no. 151 (C-584) ,12 April 1989 & JP,A,63 309591 (NIPPON DENSO CO LTD;OTHERS: 01) 16 December 1988,**

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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0001] This invention relates to grease compositions having low noise properties and high dropping points.

2. Description of the Related Art

[0002] The bearings in modern automobiles and electric appliances are typically sealed bearings. Greases for sealed bearings must provide lubrication for the life of the bearing which places high demands on greases used in such service. Sealed bearing greases must meet numerous performance requirements including extending bearing life, high temperature performance, high dropping point, low oil separation, oxidation stability, fretting wear protection and low noise.

[0003] Recently, lithium complex soap based greases have been used in sealed bearing applications. Such greases offer long service at high temperatures but suffer from generally higher noise than simple lithium soap based greases.

[0004] It would be desirable to have a sealed bearing grease containing a lithium complex soap which does not cause high noise in sealed bearings. For high temperature service improvement in grease dropping point is also desirable.

SUMMARY OF THE INVENTION

[0005] This invention relates to a grease composition having improved low noise properties and a dropping point of greater than 270°C according to standard test ASTM D566, which composition comprises:

a) a major amount of a lubricating base oil;

b) from 2 to 30 wt%, based on the grease composition, of a lithium soap complex thickener; and

c) from 0.1 to 10 wt%, based on the grease composition, of a polyalkenyl succinic anhydride, a polyalkenyl succinic acid, or a mixture thereof, wherein the polyalkenyl group has a number average molecular weight of from 300 to 4,000.

[0006] The greases according to the invention provide long bearing life and high dropping point while providing low bearing noise.

DETAILED DESCRIPTION OF THE INVENTION

[0007] A wide variety of lubrication base oils can be employed in preparing the grease compositions of the present invention. Accordingly, the lubricating oil base can be any of the conventionally used mineral oils, synthetic hydrocarbon oils or synthetic ester oils, or mixtures thereof depending upon the particular grease being prepared. In general these lubricating oils will have a viscosity in the range of about 5 to about 400 cSt at 40°C, although typical applications will require an oil having a viscosity ranging from about 10 to about 200 cSt at 40°C. Mineral lubricating oil base stocks used in preparing the greases can be any conventionally refined base stocks derived from paraffinic, naphthenic and mixed base crudes. Synthetic lubricating oils that can be used include esters of glycols such as a C_{13} oxo acid diester of tetraethylene glycol, or complex esters such as one formed from 1 mole of sebacic acid and 2 moles of tetraethylene glycol and 2 moles of 2-ethylhexanoic acid. Other synthetic oils that can be used include synthetic hydrocarbons such as polyalphaolefins; alkyl benzenes, e.g., alkylate bottoms from the alkylation of benzene with tetrapropylene, or the copolymers of ethylene and propylene; silicone 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 C_8 oxo alcohol with ethyl carbonate to form a half ester followed by reaction of the latter with tetraethylene glycol, etc. Other suitable synthetic oils include the polyphenyl ethers, e.g., those having from about 3 to 7 ether linkages and about 4 to 8 phenyl groups. The amount of lubricating oil in the grease can also vary broadly, but, typically, will range from about 50 wt% to about 98 wt%, preferably from about 75 wt% to about 95 wt%, of the grease.

[0008] The grease composition contains a thickener dispersed in the lubricating base oil to form a grease. Preferred thickeners are lithium soap complex thickeners containing a lithium soap of a C_{12} to C_{24} hydroxy fatty acid and a second lithium salt compound which is a lithium salt of a C_2 to C_{12} aliphatic or cycloaliphatic dicarboxylic acid, a lithium salt of a C_3 to C_{14} hydroxy carboxylic acid which has the hydroxy group separated from the carboxyl group by six or less

carbon atoms or a mixture thereof. The lithium salt of boric acid may be added as a third thickener component. Preferred hydroxy fatty acids include hydroxystearic, hydroxyricinoleic, hydroxybehenic and hydroxypalmitic. Especially preferred is 12-hydroxystearic acid. The second lithium salt compound is preferably a C_6 to C_{10} aliphatic dicarboxylic acid, more preferably azelaic or sebacic acids, especially azelaic acid. The hydroxy-carboxylic acid is preferably lactic acid, salicylic acid or other hydroxy-benzoic acids, more preferably salicylic acid. The amount of lithium soap complex thickeners is preferably from 5 to 20 wt%, based on grease. The ratio of hydroxy fatty acid to aliphatic dicarboxylic acid and/or hydroxy-carboxylic acid is from 10:0.5 to 10:15, preferably 10:1.5 to 10:6.

[0009] The grease composition will also contain a polyalkenyl succinic anhydride, a polyalkenyl succinic acid or mixture thereof as dispersant. The polyalkenyl group will have a number average molecular weight of from 300 to 4,000, preferably 400 to 2,500. Preferred polyalkenyl groups are poly-C_3 and poly-C_4 olefins, especially polyisobutylene. Polyisobutylene succinic anhydride and/or acid will hereinafter be collectively referred to as PIBSA. The amount of dispersant is preferably from 0.1 wt% to 5 wt%, based on grease.

[0010] Lithium complex soap greases are prepared by heating hydroxy fatty acid and base oil, adding a concentrated aqueous solution of lithium hydroxide. Heating is continued at elevated temperature to remove most of the water. The reaction mixture is then cooled to about 100°C and dicarboxylic acid is added followed by a second portion of concentrated lithium hydroxide. This mixture is again heated to elevated temperature to again remove water. After heating is completed, the grease mixture is cooled, remaining oil and any other additives added and the grease is worked up using conventional techniques. The preparation may also be accomplished in a single step process by heating, base oil, fatty acid and dicarboxylic acid, then adding lithium hydroxide solution followed by dehydration and adding remaining oil.

[0011] The grease composition may contain small amounts of supplemental additives which include, but are not limited to, anti-corrosive agents, extreme pressure antiwear agents, pour point depressants, tackiness agents, oxidation inhibitors, dyes and the like, which are incorporated for specific purposes. The total amount of these additives will typically range from about 2 wt% to about 10 wt% based on total weight of the grease composition.

[0012] This invention will be further understood by reference to the following examples which include a preferred embodiment of the invention.

Example 1

[0013] This example illustrates the preparation of a low noise grease. A laboratory kettle was charged with 700 g of refined paraffinic base oil having a viscosity of 90 cSt at 40°C, 139.2 g of 12-hydroxystearic acid, 69.5 g of azelaic and 12.0 g of PIBSA having a polyisobutenyl radical of number average molecular weight of 950. The resulting mixture was heated with stirring until the temperature reached 90°C. At this point, heating was stopped and 345 g of a 15% solution of lithium hydroxide (heated to 90°C) was added with stirring. When addition of the lithium hydroxide solution was complete, heating was resumed to remove water. An additional 100 g of oil was added during the dehydration process to control grease hardening. Heating was continued to a temperature of 210°C at which time heating was discontinued and an additional 128 g of oil was added. The heated mixture was allowed to cool slowly to a temperature of 70°c or less. The cooled greases was milled at 0.005 inch clearance and deaerated. The grease samples described in Table 1 of Example 3 were manufactured using this procedure. Component amounts were varied as described in Samples 1-3 and Comparative Samples 4-14.

Example 2

[0014] Grease noise was measured according to the following procedure which is more fully described in Japanese Patent 53-2357. Solid particles in grease result in vibrations which can be converted into an electrical signal. After correcting for race noise of bearings under standard conditions, the electrical signal due to solid particles including soap particles in grease can be measured according to the following procedure. A vibration detection device is attached to the outer ring of the bearings. The detection device converts the vibrations into an electrical signal which is amplified and sent to a zone wave filter. The zone wave filter compares the amplified signal received from the bearings with the known vibration frequency from the bearings alone, filters out the vibration frequencies from the bearings and sends the filtered electronic signal through an amplifier to a clipper circuit. The clipper circuit "clips" the electrical signal from the amplifier at a given level. This level is set so as to measure only peak voltages which correspond to solid particle noise from particles in the grease. The clipping level is set at a value greater than the peak value corresponding to the race noise from the bearings.

Example 3

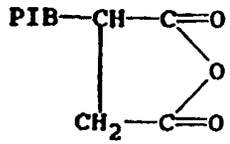
[0015] This example compares the noise properties and dropping point of a grease formulated with a polyisobutenyl

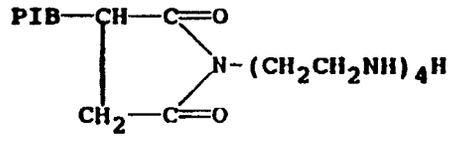
succinic anhydride/acid dispersant according to the invention with polyisobutenyl succinic imide type dispersants described in Japanese Patent A2 58125794. Greases were formulated using the procedures of Example 1. Noise was tested using the procedure of Example 2 and dropping point was measured according to test method ASTM D566. The results are set forth in Table 1.

TABLE 1

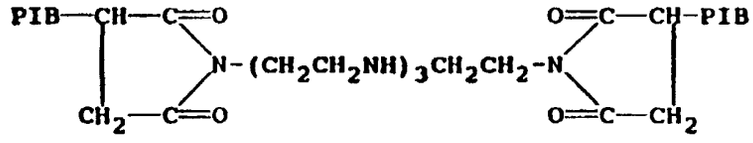
Composition (Wt% Based On Grease)	Samples 1	2	3	Comparative Samples 4	5	6	7	8	9	10	11	12	13	14(6)
12-hydroxystearic acid	11.6	----------	----------	----------	----------	----------	same	----------	----------	----------	----------	----------	----------	-
Azelaic acid	5.8	----------	----------	----------	----------	----------	same	----------	----------	----------	----------	----------	----------	-
Lithium hydroxide	4.3	4.3	4.4	4.2	----------	----------	----------	----------	same	----------	----------	----------	4.3	-
Dispersant A (1)	1.0	1.5	2.0	-	-	-	-	-	-	-	-	-	-	-
Dispersant B (2)				-	1.5	2.0	-	-	-	-	-	-	-	-
Dispersant C (3)				-	-	-	1.0	1.5	2.0	-	-	-	-	-
Dispersant D (4)				-	-	-	-	-	-	1.5	2.0	2.5	3.0	-
Property														
Worked consistency (ASTM D217)	242	229	263	260	249	239	234	239	252	255	252	224	221	235
Dropping point, °C (ASTM D566)	>270	>270	>270	234	264	247	204	241	208	239	257	270	255	>270
Noise (5)	452	128	379	7,808	480	372	3,255	326	562	599	517	83	100	2,725

Notes:

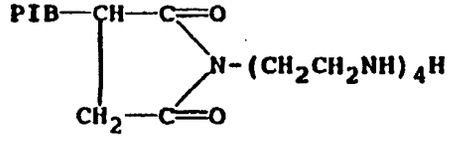
(1) 80 wt% solution of  in refined mineral oil; average molecular weight of PIB is 950

(2) 50 wt% solution of 

in refined mineral oil, average molecular weight of PIB is 950

(3) 52 wt% solution of 

in refined mineral oil, average molecular weight of PIB is 950

(4) 60 wt% solution of borated 

in refined mineral oil, average molecular weight of PIB is 950

(5) Counts/120 second as determined by method of Example 2

(6) Commercial sample of lithium complex grease

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[0016] Greases formulated with a polyisobutenyl succinic anhydride/ acid type dispersant (Samples 1-3) showed significantly better noise property and higher dropping point compared to a lithium complex grease formulated without dispersant (Sample 4). Samples 1-3 also demonstrated consistently higher dropping points compared to greases formulated with polyisobutenyl succinic imide type dispersants (Comparative Samples 5-13). A commercial lithium complex grease (Sample 14) demonstrated a much inferior noise property compared to Samples 1-3 according to this invention.

Claims

1. A grease composition having improved low noise properties and a dropping point of greater than 270°C according to standard test ASTM D566, which composition comprises:

   a) a major amount of a lubricating base oil;

   b) from 2 to 30 wt%, based on the grease composition, of a lithium soap complex thickener, and

   c) from 0.1 to 10 wt%, based on the grease composition, of a polyalkenyl succinic anhydride, a polyalkenyl succinic acid, or a mixture thereof, wherein the polyalkenyl group has a number average molecular weight of from 300 to 4,000.

2. The grease composition of claim 1 wherein the lithium soap complex thickener contains a lithium soap of a C_{12} to C_{24} hydroxy fatty acid and a lithium salt of a C_2 to C_{12} aliphatic or cycloaliphatic dicarboxylic, a lithium salt of a C_3-C_{14} hydroxy carboxylic acid, or a mixture thereof.

3. The grease composition of claim 2 wherein the lithium soap complex thickener contains a lithium soap of 12-hydroxystearic acid and a lithium salt of azelaic acid and/or lithium salt of salicylic acid.

4. The grease composition of claim 2 or 3 wherein the ratio of hydroxy fatty acid to aliphatic or cycloaliphatic dicarboxylic acid and/or hydroxy carboxylic acid is from 10:0.5 to 10:15.

5. The grease composition of any preceding claim wherein the polyalkenyl moiety in the polyisobutenyl succinic anhydride and/or polyisobutenyl succinic acid has a number average molecular weight of from 400 to 2,500.

6. A method for improving noise reduction and providing a dropping point of greater than 270°C (ASTM D566) in a grease which comprises incorporating into a base oil from 2 to 30 wt%, based on grease, of a lithium soap complex thickener and from 0.1 to 10 wt%, based on grease, of a polyisobutenyl succinic anhydride, a polyisobutenyl succinic acid or mixture thereof wherein the polyalkenyl group has a number average molecular weight of from 300 to 4,000.

7. Use of a polyalkenyl succinic anhydride, a polyalkenyl succinic acid, or a mixture thereof wherein the polyalkenyl group has a number average molecular weight of 300 to 4000 in an amount from 0.1 to 10wt%, based on grease, in a grease composition containing from 2 to 30 wt%, based on grease, of a lithium soap complex thickener, to improve the noise properties of the grease composition.

8. Use of a polyalkenyl succinic anhydride, a polyalkenyl succinic acid, or a mixture thereof wherein the polyalkenyl group has a number average molecular weight of 300 to 4000 in an amount from 0.1 to 10wt%, based on grease, in a grease composition containing from 2 to 30 wt%, based on grease, of a lithium soap complex thickener, to improve the dropping point of the grease composition.

Patentansprüche

1. Schmierfettzusammensetzung mit verbesserten Lärmverminderungseigenschaften und einem Tropfpunkt von mehr als 270°C gemäß dem Standardtest ASTM D566, wobei die Zusammensetzung

   a) eine größere Menge eines Schmierbasisöls,
   b) 2 bis 30 Gew.%. bezogen auf die Schmierfettzusammensetzung, von einem Lithiumseifenkomplex-Verdik-

kungsmittel und
c) 0,1 bis 10 Gew.%, bezogen auf die Schmierfettzusammensetzung, von einem Polyalkenylbernsteinsäureanhydrid, einer Polyalkenylbernsteinsäure oder einer Mischung davon, wobei die Polyalkenylgruppe ein durchschnittliches Molekulargewicht (Zahlenmittel) von 300 bis 4000 hat,

enthält.

2. Schmierfettzusammensetzung nach Anspruch 1, bei der das Lithiumseifenkomplex-Verdickungsmittel eine Lithiumseife einer C_{12}- bis C_{24}-Hydroxyfettsäure und ein Lithiumsalz einer aliphatischen oder cycloaliphatischen C_2- bis C_{12}- Dicarbonsäure, ein Lithiumsalz einer C_3- bis C_{14}-Hydroxycarbonsäure oder eine Mischung davon enthält.

3. Schmierfettzusammensetzung nach Anspruch 2, bei der das Lithiumseifenkomplex-Verdickungsmittel eine Lithiumseife von 12-Hydroxystearinsäure und ein Lithiumsalz von Azelainsäure und/oder Lithiumsalz von Salicylsäure enthält.

4. Schmierfettzusammensetzung nach Anspruch 2 oder 3, bei der das Verhältnis von Hydroxyfettsäure zu aliphatischer oder cycloaliphatischer Dicarbonsäure und/oder Hydroxycarbonsäure 10:0,5 bis 10:15 beträgt.

5. Schmierfettzusammensetzung nach einem der vorhergehenden Ansprüche, bei der die Polyalkenyleinheit in dem Polyisobutenylbernsteinsäureanhydrid und/oder der Polyisobutenylbernsteinsäure ein durchschnittliches Molekulargewicht (Zahlenmittel) von 400 bis 2500 hat.

6. Verfahren zur Verbesserung der Lärmverminderung und zur Lieferung eines Tropfpunkts von mehr als 270°C (ASTM D566) bei einem Schmierfett, bei dem in ein Basisöl 2 bis 30 Gew.%, bezogen auf das Schmierfett, von einem Lithiumseifenkomplex-Verdickungsmittel und 0,1 bis 10 Gew.%, bezogen auf das Schmierfett, von einem Polyisobutenylbernsteinsäureanhydrid, einer Polyisobutenylbernsteinsäure oder einer Mischung davon eingebracht wird, wobei die Polyalkenylgruppe ein durchschnittliches Molekulargewicht (Zahlenmittel) von 300 bis 4000 hat.

7. Verwendung von einem Polyalkenylbernsteinsäureanhydrid, einer Polyalkenylbernsteinsäure oder einer Mischung davon, wobei die Polyalkenylgruppe ein durchschnittliches Molekulargewicht (Zahlenmittel) von 300 bis 4000 hat, in einer Menge von 0,1 bis 10 Gew.%, bezogen auf das Schmierfett, in einer Schmierfettzusammensetzung, die 2 bis 30 Gew.%, bezogen auf das Schmierfett, eines Lithiumseifenkomplex-Verdikkungsmittels enthält, zur Verbesserung der Lärmverminderungseigenschaften der Schmierfettzusammensetzung.

8. Verwendung von einem Polyalkenylbernsteinsäureanhydrid, einer Polyalkenylbernsteinsäure oder einer Mischung davon, wobei die Polyalkenylgruppe ein durchschnittliches Molekulargewicht (Zahlenmittel) von 300 bis 4000 hat, in einer Menge von 0,1 bis 10 Gew.%, bezogen auf das Schmierfett, in einer Schmierfettzusammensetzung, die 2 bis 30 Gew.%, bezogen auf das Schmierfett, eines Lithiumseifenkomplex-Verdikkungsmittels enthält, zur Verbesserung des Tropfpunkts der Schmierfettzusammensetzung.

Revendications

1. Composition de graisse ayant de meilleures propriétés de faible niveau de bruit et un point de goutte supérieur à 270°C selon le test standard ASTM D566, ladite composition comprenant :

(a) une quantité majeure d'une huile de base lubrifiante;
(b) 2 à 30% en poids, par rapport à la composition de graisse, d'un épaississant complexe de savon de lithium; et
(c) 0,1 à 10% en poids, par rapport à la composition de graisse, d'un anhydride polyalcénylsuccinique, d'un acide polyalcénylsuccinique ou d'un de leurs mélanges, où le groupe polyalcényle a un poids moléculaire moyen en nombre de 300 à 4000.

2. Composition de graisse selon la revendication 1, dans laquelle l'épaississant complexe de savon de lithium contient un savon de lithium d'un hydroxyacide gras en C_{12}-C_{24} et un sel de lithium d'un acide dicarboxylique aliphatique ou cycloaliphatique en C_2-C_{12}, un sel de lithium d'un acide hydroxycarboxylique en C_3-C_{14}, ou un de leurs mélanges.

3. Composition de graisse selon la revendication 2, dans laquelle l'épaississant complexe de savon de lithium contient un savon de lithium d'acide 12-hydroxystéarique et un sel de lithium d'acide azélaïque et/ou un sel de lithium d'acide salicylique.

4. Composition de graisse selon la revendication 2 ou 3, dans laquelle le rapport de l'hydroxyacide gras à l'acide dicarboxylique aliphatique ou cycloaliphatique et/ou à l'acide hydroxycarboxylique est de 10:0,5 à 10:15.

5. Composition de graisse selon l'une quelconque des revendications précédentes, dans laquelle le radical polyalcényle de l'anhydride polyisobuténylsuccinique et/ou de l'acide polyisobuténylsuccinique a un poids moléculaire moyen en nombre de 400 à 2500.

6. Procédé d'amélioration de la réduction du bruit et de l'obtention d'un point de goutte supérieur à 270°C (ASTM D566) dans une graisse, comprenant l'incorporation à une huile de base de 2 à 30% en poids, par rapport à la graisse, d'un épaississant complexe de savon de lithium et de 0,1 à 10% en poids, par rapport à la graisse, d'un anhydride polyisobuténylsuccinique, d'un acide polyisobuténylsuccinique ou d'un de leurs mélanges, où le groupe polyalcényle a un poids moléculaire moyen en nombre de 300 à 4000.

7. Utilisation d'un anhydride polyalcénylsuccinique, d'un acide polyalcénylsuccinique ou d'un de leurs mélanges, dans laquelle le groupe polyalcényle a un poids moléculaire moyen en nombre de 300 à 4000, en quantité de 0,1 à 10% en poids, par rapport à la graisse, dans une composition de graisse contenant 2 à 30% en poids, par rapport à la graisse, d'un épaississant complexe de savon de lithium, pour améliorer les propriétés de bruit de la composition de graisse.

8. Utilisation d'un anhydride polyalcénylsuccinique, d'un acide polyalcénylsuccinique ou d'un de leurs mélanges, dans laquelle le groupe polyalcényle a un poids moléculaire moyen en nombre de 300 à 4000, en quantité de 0,1 à 10% en poids, par rapport à la graisse, dans une composition de graisse contenant 2 à 30% en poids, par rapport à la graisse, d'un épaississant complexe de savon de lithium, pour améliorer le point de goutte de la composition de graisse.