

DESCRIPTION

Title of Invention

GREASE COMPOSITION

5 Technical Field

[0001]

The invention relates to a grease composition that utilizes a metal soap-based thickener.

Background Art

10 [0002]

Grease has been mainly used for slide bearings, rolling bearings, and sliding surfaces where it is difficult to maintain adhesion of a lubricant film due to the movement of the contact surface. Metal soap grease that utilizes a metal soap-based thickener exhibits excellent water resistance, heat resistance, and mechanical stability. In particular, lithium soap-based grease has been most widely used as universal grease, and has been used for bearings, gears, ball joints, pinions, and the like which have steel-resin sliding parts.

[0003]

20 A grease composition that includes silicone oil, a lithium soap thickener, 3 to 25 mass% of a polytetrafluoroethylene resin powder, and 1 to 15 mass% of a saturated fat acid amide has been proposed as resin grease used for steel-resin sliding parts (see PTL 1).

25 However, since the grease composition disclosed in PTL 1 utilizes silicone oil, namely a high-density base oil, the dispersibility of the solid lubricant is poor. Moreover, the friction coefficient cannot be sufficiently decreased, and a stick-slip phenomenon easily occurs.

Citation List

Patent Literature

[0004]

PTL 1: JP-A-2011-225781

Summary of Invention

5 Technical Problem

[0005]

An object of the invention is to provide a grease composition that improves the dispersibility of a solid lubricant, that provides a sufficiently low friction coefficient in steel-resin sliding parts, and that prevents a stick-slip phenomenon.

10 Solution to Problem

[0006]

The inventors of the invention conducted extensive studies in order to achieve the above object. As a result, the inventors found that a grease composition that improves the dispersibility of a solid lubricant, that provides a sufficiently low friction
15 coefficient in steel-resin sliding parts, and that prevents a stick-slip phenomenon can be obtained, by utilizing a lubricant base oil having a specific density.

The invention was completed based on the above finding, and provides the following.

[0007]

- 20 (1) A grease composition comprising a lubricant base oil having a density of 0.75 to 0.95 g/cm³ at 15°C, an amide compound, a solid lubricant, and a metal soap-based thickener.
- (2) The grease composition according to (1), wherein the solid lubricant is a layered compound or a fluororesin.
- 25 (3) The grease composition according to (1) or (2), wherein the amide compound is a monoamide or a bisamide.

Advantageous Effects of Invention

[0008]

The grease composition according to the invention ensures that the solid lubricant is sufficiently dispersed therein, provides a low friction coefficient in steel-resin sliding parts, and prevents a stick-slip phenomenon.

5 **Description of Embodiments**

[0009]

The grease composition according to the invention comprises a lubricant base oil having a density of 0.75 to 0.95 g/cm³ at 15°C, an amide compound, a solid lubricant, and a metal soap-based thickener.

10 [Lubricant base oil]

A mineral oil-based lubricant base oil or a synthetic lubricant base oil may be used as the lubricant base oil used in connection with the invention as long as the density is 0.75 to 0.95 g/cm³ at 15°C. If the density of the lubricant base oil falls outside the above range, the dispersibility of the solid lubricant may decrease, and the
15 friction coefficient may not be sufficiently decreased. The density of the lubricant base oil is more preferably 0.8 to 0.9 g/cm³.

It is preferable that the lubricant base oil have a kinematic viscosity of 1 to 500 mm²/s at 40°C, and more preferably 5 to 100 mm²/s. If the kinematic viscosity of the lubricant base oil falls outside the range of 1 to 500 mm²/s at 40°C, it may be difficult to
20 easily prepare a grease composition having the desired consistency. In order to prepare grease having excellent lubricity, it is preferable to use a lubricant base oil having a viscosity index of 90 or more in particular, 95 to 250, a pour point of -10°C or less in particular, -15 to -70°C, and a flash point of 150°C or more.

[0010]

25 Examples of the mineral oil-based lubricant base oil include lubricant fractions obtained by distilling crude oil under atmospheric pressure optionally followed by distillation under reduced pressure to obtain a distillate, and refining the distillate using

various types of refining process. Examples of the refining process include hydrotreating, solvent extraction, solvent dewaxing, hydrodewaxing, washing with sulfuric acid, clay treatment, and the like. The base oil used in connection with the invention can be obtained by combining these processes in an appropriate order. A mixture of a plurality of refined oils having different properties is also useful, wherein the mixture is obtained by using different types of crude oils or distillates and by a different combination and/or order of processes. The base oil obtained by each method may preferably be used as long as the properties of the base oil are adjusted to satisfy the above density range.

[0011]

It is preferable to use a material that exhibits excellent hydrolytic stability as the synthetic lubricant base oil. Examples of the synthetic lubricant base oil include polyolefins such as a poly- α -olefin, a polybutene, and a copolymer of two or more olefins, polyesters, polyalkylene glycols, alkylbenzenes, alkyl-naphthalenes, and the like. It is preferable to use a poly- α -olefin from the viewpoint of availability, cost, viscosity, oxidation stability, and compatibility with a system member. A polymer of 1-dodecene or 1-decene is more preferable as the poly- α -olefin from the viewpoint of cost.

[0012]

These synthetic lubricant base oils may be used either alone or in combination. The synthetic lubricant base oil may be used in combination with the mineral oil-based lubricant base oil.

When using a mixture of a plurality of types of lubricant base oil including a synthetic lubricant base oil, the properties of each base oil are not necessary to fall within the above range as long as the base oil mixture satisfies the above properties. Therefore, each synthetic base oil need not necessarily satisfy the above properties, but it is preferable that the properties of each synthetic base oil fall within the above ranges.

The content of the lubricant base oil in the grease composition is preferably 50

to 95 mass%, and more preferably 60 to 85 mass%, based on the total amount of the grease composition. If the content of the lubricant base oil is outside the range of 50 to 95 mass%, it may be difficult to easily prepare a grease composition having the desired consistency.

5 [0013]

[Amide compound]

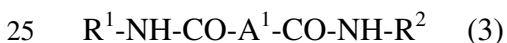
The amide compound used in connection with the invention is a compound that includes at least one amide group (-NH-CO-). A compound that includes one amide group (monoamide), a compound that includes two amide groups (bisamide), and a
10 compound that includes three amide groups (triamide) may be used as the amide compound. The bisamide and the triamide have an advantage in that the frictional resistance in the sliding part can be reduced even when the amide compound is used in a relatively small amount. Therefore, the bisamide and the triamide are most suitable as the amide compound.

15 The bisamide may be an acid amide of a diamine or an acid amide of a diacid.

It is preferable to use an amide compound having a melting point of 40 to 180°C (more preferably 80 to 180°C, and still more preferably 100 to 170°C) and a molecular weight of 242 to 932 (more preferably 298 to 876).

The monoamide is represented by the following general formula (1), the
20 bisamide is represented by the following general formulas (2) and (3), and the triamide is represented by the following general formula (4).

[0014]



[0015]

wherein R^1 , R^2 , and R^3 are independently a hydrocarbon group having 5 to 25 carbon atoms. The hydrocarbon group may be an aliphatic hydrocarbon group, an alicyclic hydrocarbon group, or an aromatic hydrocarbon group. R^2 in the general formula (1) may be a hydrogen atom. A^1 , A^2 , and A^3 are independently an aliphatic hydrocarbon group having 1 to 10 carbon atoms, an alicyclic hydrocarbon group, an aromatic hydrocarbon group, or a divalent hydrocarbon group having 1 to 10 carbon atoms formed by combining these groups, and M is an amide group.

It is preferable that R^2 is a hydrogen atom or a saturated or unsaturated chain hydrocarbon group having 10 to 20 carbon atoms when the amide compound is the monoamide.

It is preferable that A^1 is a divalent saturated chain hydrocarbon group having 1 to 4 carbon atoms when the amide compound is the acid amide of a diamine.

Some of the hydrogen atoms of the hydrocarbon group represented by R^1 , R^2 , or A^1 in the general formulas (2) and (3) may be substituted with a hydroxyl group (-OH).

[0016]

An amide compound in which A^1 , A^2 , and A^3 are an aliphatic hydrocarbon group is referred herein to as “aliphatic amide”, an amide compound in which at least one of A^1 , A^2 , and A^3 is an aromatic hydrocarbon group is referred herein to as “aromatic amide”, and an amide compound in which at least one of A^1 , A^2 , and A^3 is an alicyclic hydrocarbon group or an aromatic hydrocarbon group is referred herein to as “non-aliphatic amide”.

It is preferable that R^1 , R^2 , and R^3 is a saturated or unsaturated chain hydrocarbon group having 10 to 20 carbon atoms when the amide compound is the aliphatic amide.

It is preferable that R^1 , R^2 , and R^3 is a saturated or unsaturated chain hydrocarbon group having 10 to 20 carbon atoms or an aromatic hydrocarbon group when the amide compound is the aromatic amide.

The non-aliphatic amide may also be used as the amide compound, but it is preferable to use the aliphatic amide as the amide compound. It is preferable that A¹ is a divalent saturated chain hydrocarbon group having 1 to 4 carbon atoms when the amide compound is the acid amide of a diamine (general formula (3)).

5 [0017]

Specific examples of the monoamide include saturated fatty acid amides such as lauric acid amide, palmitic acid amide, stearic acid amide, behenic acid amide, and hydroxystearic acid amide, unsaturated fatty acid amides such as oleic acid amide and erucic acid amide, substituted amides of a saturated or unsaturated long-chain fatty acid
10 and a long-chain amine such as stearylstearyl acid amide, oleyloleic acid amide, oleylstearyl acid amide, and stearyloleic acid amide, and the like.

[0018]

Specific examples of the acid amide of a diamine represented by the general formula (2) include ethylene bis-stearyl acid amide, ethylene bis-isostearyl acid amide,
15 ethylene bis-oleic acid amide, methylene bis-lauric acid amide, hexamethylene bis-oleic acid amide, hexamethylene bis-hydroxystearic acid amide, and the like. Specific examples of the bisamide of a diacid represented by the general formula (3) include N,N'-bis-stearylsebacic acid amide and the like.

Among these, the amide compounds represented by the general formula (2) or
20 (3) in which R¹ and R² are independently a saturated chain hydrocarbon group or an unsaturated chain hydrocarbon group having 12 to 20 carbon atoms are preferable.

[0019]

There are various triamide compounds that are represented by the general formula (4). Specific examples of a compound among the compounds represented by
25 the general formula (4) that can be suitably used in connection with the invention include an N-acylamino acid diamide compound. The N-acyl group included in the N-acylamino acid diamide compound is preferably a linear or branched saturated or

unsaturated aliphatic acyl group having 1 to 30 carbon atoms, or an aromatic acyl group in particular, a caproyl group, a capryloyl group, a lauroyl group, a myristoyl group, or a stearoyl group. The amino acid included in the N-acylamino acid diamide compound is preferably aspartic acid or glutamic acid. The amine of the amide group included in the N-acylamino acid diamide compound is preferably a linear or branched saturated or unsaturated aliphatic amine having 1 to 30 carbon atoms, and more preferably butylamine, octylamine, laurylamine, isostearylamine, or stearylamine. In particular, N-lauroyl-L-glutamic acid- α,γ -di-n-butylamide is preferable.

[0020]

These amide compounds may be used either alone or in combination. The content of the amide compound in the grease composition is preferably 1 to 50 mass%, and more preferably 5 to 30 mass%, based on the total amount of the grease composition.

When the amide compound is heated and melted in the presence of the lubricant base oil, the lubricant base oil is held by the amide compound that forms a three-dimensional network structure. Therefore, the friction coefficient in steel-resin sliding parts further decreases, in comparison with the case of merely dispersing and mixing the amide compound in grease. Moreover, a stick-slip phenomenon does not occur.

[0021]

[Solid lubricant]

The solid lubricant is not particularly limited as long as the solid lubricant is normally used as a lubricant. It is preferable to use a layered compound or a fluororesin as the solid lubricant due to excellent lubricity.

The term "layered compound" used herein refers to a compound having a layered crystal structure. For example, melamine cyanurate, molybdenum disulfide, boron nitride, graphite, mica, fluorinated graphite, and the like are preferable as the

layered compound.

Examples of a preferable fluoro-resin include a polytetrafluoroethylene (PTFE), a tetrafluoroethylene-perfluoroalkyl vinyl ether copolymer (PFA), a tetrafluoroethylene-hexafluoropropylene copolymer (FEP), a tetrafluoroethylene-ethylene copolymer (ETFE), a polyvinylidene fluoride (PVDF), a polychlorotrifluoroethylene (PCTFE), and the like.

These solid lubricants may be used either alone or in combination. A solid lubricant having an appropriate particle size may be selected depending on the application. It is preferable to use a solid lubricant having a particle size (diameter) of 0.2 to 50 μm , and more preferably 1 to 10 μm .

The content of the solid lubricant in the grease composition is preferably 0.1 to 10 mass%, and more preferably 0.2 to 5 mass%, based on the total amount of the grease composition.

[0022]

[Metal soap-based thickener]

The metal soap-based thickener is a thickener that includes a metal carboxylate. The carboxylic acid may be a carboxylic acid derivative that includes a hydroxyl group or the like.

The carboxylic acid may be either an aliphatic carboxylic acid such as stearic acid and azelaic acid or an aromatic carboxylic acid such as terephthalic acid. An aliphatic monocarboxylic acid or dicarboxylic acid in particular, an aliphatic carboxylic acid having 6 to 20 carbon atoms may be used. It is preferable to use an aliphatic monocarboxylic acid having 12 to 20 carbon atoms or an aliphatic dicarboxylic acid having 6 to 14 carbon atoms. An aliphatic monocarboxylic acid that includes one hydroxyl group is preferable.

The metal may be an alkali metal such as lithium and sodium, an alkaline-earth metal such as calcium, or an amphoteric metal such as aluminum. An alkali metal in

particular, lithium is preferably used.

[0023]

The thickener may be added in the form of metal soap. The carboxylic acid and a metal source (e.g., metal salt and metal salt hydroxide) may also be separately
5 added and reacted to produce a metal soap thickener, when preparing grease.

Such metal carboxylates may be used either alone or in combination. For example, it is particularly preferable to use a mixture of lithium 12-hydroxystearate and lithium azelate.

The content of the metal soap-based thickener in the grease composition may be
10 appropriately determined as long as the desired consistency can be obtained. For example, the content of the metal soap-based thickener in the grease composition is preferably 2 to 30 mass%, and more preferably 5 to 15 mass%, based on the total amount of the grease composition.

[0024]

15 [Additive]

The grease composition according to the invention may optionally include a detergent, a dispersant, an antiwear agent, a viscosity index improver, an antioxidant, an extreme pressure agent, a rust-preventive agent, a corrosion inhibitor, and the like that are normally used for a lubricant or grease in addition to the above components.

20 [0025]

[Preparation method]

The grease composition according to the invention may be prepared using a normal grease preparation method. It is preferable to heat a mixture comprising the amide compound to a temperature equal to or higher than the melting point of the amide
25 compound at least once, after mixing the amide compound.

Specifically, the grease composition may be prepared by heating the amide compound and the lubricant base oil to a temperature equal to or higher than the melting

point of the amide compound, cooling the mixture, and then physically mixing the cooled mixture with normal grease that comprises the solid lubricant, the thickener, and the lubricant base oil. Alternatively, all of the components including the thickener may be mixed, heated to a temperature equal to or higher than the melting point of the amide compound, and then cooled.

[0026]

[Lubrication target]

The grease composition according to the invention may suitably be used for lubrication between various resin sliding members and various metal sliding members.

Examples of the resin sliding member include sliding members formed of a nylon resin, a polycarbonate (PC) resin, a polybutylene terephthalate (PBT) resin, a polyacetal (POM) resin, and the like having a long-term heat resistant temperature (UL standard) of 50 to 150°C. In particular, the grease composition is suitable for a member where a nylon 6 (PA6) resin is used. Examples of the metal sliding member include sliding members formed of bearing steel, carbon steel, stainless steel (SUS), and the like. In particular, the grease composition is suitable for a member where bearing steel SUJ-2 is used.

Examples

[0027]

1. Lubricant base oil

(1) Mineral oil-based lubricant base oil

- Lubricant base oil obtained by distilling atmospheric distillation residue under reduced pressure and subjecting the resulting distillate to solvent refining

Kinematic viscosity at 40°C: 68 mm²/s

Density at 15°C: 0.87 g/cm³

Viscosity index: 100

Pour point: -10°C

Flash point: 250°C

[0028]

(2) Synthetic lubricant base oil

(a) Poly- α -olefin (“Durasyn 170” manufactured by INEOS)

5 Kinematic viscosity at 40°C: 68 mm²/s

Density at 15°C: 0.83 g/cm³

Viscosity index: 133

Pour point: -45°C

Flash point: 250°C

10 (b) Polydimethylsiloxane (silicone oil; “KF-96” manufactured by Shin-Etsu Chemical Co., Ltd., 50 cs, 100 cs)

Kinematic viscosity at 40°C: 68 mm²/s

Density at 15°C: 0.96 g/cm³

(c) Linear perfluoro polyether (fluorinated oil; “FOMBLIN M15” manufactured by
15 Solvay Solexis)

Kinematic viscosity at 40°C: 85 mm²/s

Density at 15°C: 1.83 g/cm³

[0029]

2. Amide compound

20 (1) Aliphatic amide

(a) Ethylene bis-stearic acid amide (special grade reagent)

(b) Ethylene bis-oleic acid amide (special grade reagent)

(c) Stearic acid monoamide (special grade reagent)

(2) Aromatic amide

25 (a) m-Xylylene bis-stearic acid amide (special grade reagent)

[0030]

3. Solid lubricant

(1) Melamine cyanurate (MCA, average particle size: 4 μm , “MELAPUR MC25” manufactured by BASF)

(2) Molybdenum disulfide (average particle size: 0.5 μm , “Nichimoly M-5 Powder” manufactured by Daizo Corporation)

5 (3) Polytetrafluoroethylene (PTFE, average particle size: 4 μm , “KTL-8N” manufactured by Kitamura Limited)

(4) Boron nitride (average particle size: 2 μm , “HP-P1” manufactured by Mizushima Ferroalloy Co., Ltd.)

(5) Silicon oxide (average particle size: 35 μm , special grade reagent)

10 The average particle size was measured by laser diffractometry.

[0031]

4. Metal soap-based thickener

(1) Lithium 12-hydroxystearate (“Lithium stearate” in Tables 1 and 2)

(2) Mixture of lithium 12-hydroxystearate and lithium azelate (mixing ratio: 2:1)

15 (“Lithium soap mixture” in Tables 1 and 2)

5. Additive

Diphenylamine was added to each composition as an antioxidant.

[0032]

[Preparation method]

20 Each component was charged into a vessel in the amount (mass%) shown in Table 1 or 2, heated to 150°C, which is a temperature equal to or higher than the melting point of the amide, stirred using a magnetic stirrer, and then cooled to room temperature. The mixture was dispersed under pressure using a roller (triple roll) to prepare a grease composition.

25 [0033]

[Evaluation method]

An evaluation test was performed using a reciprocating friction tester utilizing a

ball and a disk. A sphere made of SUJ-2 and having a diameter of 1/4 inches was used as a metal sliding member, and a plate made of nylon 6 (“N6 (NC)” manufactured by Toray Plastics Precision Co., Ltd.) was used as a resin sliding member.

The test load was set to 2000 gf, the sliding speed was set to 10 mm/s, and the amplitude was set to 20 mm. The grease was applied to the disk, and the presence or absence of a stick-slip phenomenon was evaluated from the friction coefficient and the frictional force waveform during sliding. Specifically, it was determined that a stick-slip phenomenon occurred when the frictional force during sliding in one direction was not constant.

[0034]

[Evaluation results]

When only the mineral oil and the lithium soap-based thickener were mixed, the friction coefficient was high, and a stick-slip phenomenon occurred (Comparative Example 7).

When only the mineral oil, the lithium soap-based thickener, and the solid lubricant were mixed, the friction coefficient slightly decreased, and a stick-slip phenomenon was somewhat suppressed insufficiently (Comparative Examples 1 to 6).

When the mineral oil, the lithium soap-based thickener, the solid lubricant, and the aliphatic amide were mixed, the friction coefficient further decreased, and a stick-slip phenomenon was suppressed (Examples 1 to 12).

When using the silicone oil or the fluorinated oil as the base oil, the melted amide compound and the base oil were separated into two phases and failed to form uniform dissolution even when the mixture was heated to a temperature equal to or higher than the melting point of the amide compound (Comparative Examples 8 and 9).

Therefore, silicon oxide was added to the silicone oil, and polytetrafluoroethylene was added to the fluorinated oil. However, since a heterogeneous mixture was obtained, the friction test could not be performed (Comparative Examples 10 and 11).

[0035]

[TABLE 1]

	Example	1	2	3	4	5	6	7	8	9	10	11	12	13
Lubricant base oil	Mineral oil	75	75	75	75	75	75	73		70	80	78	80.5	75
	Poly- α -olefin								75					
Amide compound	Stearic acid monoamide		15											
	Stearic acid bisamide	15			15	15	15	15	15	20	10	10	10	
	Oleic acid bisamide			15										
	Aromatic amide													15
Solid lubricant	MCA				1									
	P T F E					1								
	Boron nitride	1	1	1				1	1	1	1	3	0.5	1
	Molybdenum disulfide						1							
Thickener	Lithium stearate	8	8	8	8	8	8		8	8	8	8	8	8
	Lithium soap mixture							10						
Antioxidant	Diphenylamine	1	1	1	1	1	1	1	1	1	1	1	1	1
Evaluation results	Resin-steel BOD friction coefficient	0.050	0.055	0.053	0.055	0.050	0.054	0.050	0.048	0.045	0.055	0.052	0.053	0.055
	Resin-steel stick-slip	O	O	O	O	O	O	O	O	O	O	O	O	O

[0036]

5 [TABLE 2]

Comparative Example		1	2	3	4	5	6	7	8	9	10	11
Lubricant base oil	Mineral oil	8 8	8 3	8 8	8 8	8 8	8 6	8 9				
	Silicone oil								8 3		7 5	
	Fluorinated oil									8 3		7 5
Amide compound	Stearic acid monoamide											
	Stearic acid bisamide								1 5	1 5	1 5	1 5
	Oleic acid bisamide											
	MCA			1								
Solid lubricant	P T F E				1							8
	Boron nitride	1	1				3		1	1	1	1
	Molybdenum disulfide					1						
	Silicon oxide										8	
Thickener	Lithium stearate	1 0		1 0	1 0	1 0	1 0	1 0				
	Lithium soap mixture		1 5									
Antioxidant	Diphenylamine	1	1	1	1	1	1	1	1	1	1	1
Evaluation results	Resin-steel BOD friction coefficient	0.070	0.075	0.070	0.072	0.075	0.070	0.080	impossible	impossible	impossible	impossible
	Resin-steel stick-slip	Δ	Δ	×	Δ	Δ	Δ	×	impossible	impossible	impossible	impossible

Industrial Applicability

[0037]

Since the grease composition according to the invention ensures that the solid lubricant is sufficiently dispersed therein, provides a low friction coefficient in steel-resin sliding parts, and prevents a stick-slip phenomenon, the grease composition is useful for lubrication between a resin sliding member and a metal sliding member

5 (e.g., bearing, gear, ball joint, and pinion).

CLAIMS

[Claim 1]

5 A grease composition comprising a lubricant base oil having a density of 0.75 to 0.95 g/cm³ at 15°C, an amide compound, a solid lubricant, and a metal soap-based thickener.

[Claim 2]

10 The grease composition according to claim 1, wherein the solid lubricant is a layered compound or a fluororesin.

[Claim 3]

15 The grease composition according to claim 1, wherein the amide compound is a monoamide or a bisamide.