A grease of the invention includes: a base oil containing hydrocarbon; and diurea that is represented by a formula (1) below as a thickener and is in a range from 15 mass % to 25 mass % based on a total amount of a composition, the grease exhibiting a worked penetration in a range from 170 to 295.

\[
R_1\text{NHCONHR}_2\text{NHCONHR}_3
\]

(1)

In the formula, \(R_1\) and \(R_3\) are mutually different and represent a hydrocarbon group having 6 to 18 carbon atoms; and \(R_2\) is a divalent aromatic hydrocarbon group having 6 to 13 carbon atoms.
GREASE AND METHOD FOR SOFTENING GREASE

TECHNICAL FIELD

[0001] The present invention relates to a grease and a method for softening the grease, more specifically, low dusting grease and a method for further softening the grease.

BACKGROUND ART

[0002] A precision electronics manufacturing device such as a semiconductor manufacturing device and a liquid crystal manufacturing device requires very clean environments and, therefore, is set in a clean room. Moreover, a food manufacturing plant, a medicine manufacturing plant and the like also require clean environments in order to avoid invasion of foreign substances into products. Machinery and devices used in such environments are provided with various bearings, sliding portions and connection portions. In lubricating portions of the bearings, sliding portions and connection portions, low dusting grease is used.

[0003] As the low dusting grease, a fluorine grease is exemplarily used. However, since the fluorine grease is generally expensive and lacks of lubricity, torque of the bearings filled with the fluorine grease may be increased. Moreover, in manufacturing precision electronics, mixing of a halogen component is unfavorable. Further, low dusting characteristics are not sufficient. As low dusting grease containing no halogen, lithium soap grease and lithium complex soap grease are used. There has been exemplarily proposed a grease containing, as the thickener, 15 to 30% of a lithium salt of a hydroxy-free fatty acid having 10 or more carbon atoms, relative to a whole composition, the lithium salt being blended in a form of a fiber with 2 μm or less of each of a length and a diameter (see Patent Literature 1). Since the grease contains a metal salt, when the grease is used in a semiconductor manufacturing device and a lubricating agent is splashed, production failure may be caused. Further, low dusting characteristics are also not sufficient.

[0004] Because of the above problems, a urea grease is also used. For instance, there has been disclosed a grease containing 50% or more of an ester oil as the base oil and 20% or more of an aliphatic diurea thickener and exhibiting 220 to 300 of a worked penetration (see Patent Literature 2). Since a large amount of the ester oil is contained in the grease, hydrolysis resistance may be affected to shorten a lifetime of the grease. Further, low dusting characteristics are also not sufficient.

[0005] It is also reported that a base oil containing no ester is used in some urea greases. (see Patent Literature 3) Since such a grease has a worked penetration in an applicable range of 190 to 230 (i.e., hard), it is difficult to apply the grease to a usage part. Moreover, torque may be increased and lubricity may be decreased. Further, low dusting characteristics are also not sufficient.

CITATION LIST

Patent Literature(s)


SUMMARY OF THE INVENTION

Problems to be Solved by the Invention

[0009] As described above, in a typical urea grease, it is difficult to provide an appropriate consistency and to reduce an amount of generated dust.

[0010] An object of the invention is to provide a grease exhibiting lower dusting characteristics in view of the above related art. Further, when the grease is softened (i.e., when a consistency is increased), dusting characteristics are generally increased. Accordingly, an object of the invention is also to provide a method for reducing the dusting characteristics while increasing the consistency.

Means for Solving the Problems

[0011] In order to solve the above problem, the invention provides a grease and a method for softening the grease as described below.

[0012] [1] A grease according to an aspect of the invention includes: a base oil including hydrocarbon; and diurea that is represented by a formula (1) below as a thickener and is in a range from 15 mass % to 25 mass % based on a total amount of a composition, the grease exhibiting a worked penetration in a range from 170 to 295,

\[ R_1 \text{NHCONHR}_2 \text{NHCONHR}_3 \]  

(1)

in which \(R_1\) and \(R_3\) are mutually different and represent a hydrocarbon group having 6 to 18 carbon atoms; and \(R_2\) is a divalent aromatic hydrocarbon group having 6 to 13 carbon atoms.

[0013] [2] In the grease according to the above aspect of the invention, a worked penetration of the grease is in a range from 230 to 265.

[0014] [3] In the grease according to the above aspect of the invention, the hydrocarbon group is a saturated hydrocarbon group.

[0015] [4] In the grease according to the above aspect of the invention, the hydrocarbon group is an alkyl group or a cycloalkyl group.

[0016] [5] In the grease according to the above aspect of the invention, the cycloalkyl group is a cyclohexyl group.

[0017] [6] In the grease according to the above aspect of the invention, \(R_1\) and \(R_3\) are an octyl group or a cyclohexyl group.

[0018] [7] In the grease according to the above aspect of the invention, an average ratio of the cycloalkyl group in the diurea is in a range from 15 mol % to 85 mol % based on a total amount of the alkyl group and the cycloalkyl group as functional groups.

[0019] [8] In the grease according to the above aspect of the invention, the average ratio of the cycloalkyl group is in a range from 15 mol % to 50 mol %.

[0020] [9] In the grease according to the above aspect of the invention, the average ratio of the cycloalkyl group is in a range from 75 mol % to 85 mol %.

[0021] [10] In the grease according to the above aspect of the invention, a content of the hydrocarbon group is 90 mass % or more based on a total amount of the base oil.

[0022] [11] In the grease according to the above aspect of the invention, the hydrocarbon group is a poly-α-olefin.

[0023] [12] In the grease according to the above aspect of the invention, a kinematic viscosity at 40 degrees C. of the base oil is 400 mm²/s or less.

[0024] [13] According to another aspect of the invention, a softening method of the grease according to the above aspect
of the invention includes: stirring the grease while applying centrifugal force on the grease.

Since a predetermined amount of diurea having a specific structure is used as the thickener in the grease of the invention, an amount of generated dust is extremely small. Accordingly, the grease according to the above aspect of the invention is suitable for machinery and devices used particularly in a clean room. Further, when the grease is softened (i.e., when a consistency is increased), the amount of the generated dust is generally increased. However, by stirring the grease according to the above aspect of the invention while applying centrifugal force on the grease, the dusting characteristics of the grease can be further reduced although the consistency thereof is further increased.

DESCRIPTION OF EMBODIMENT(S)

A grease according to an exemplary embodiment (hereinafter, also referred to as “the present grease”) is provided by containing a hydrocarbon base oil and diurea having a specific structure as a thickener.

An exemplary embodiment of the invention will be described below in detail.

Base Oil

A base oil used for manufacturing the present grease contains hydrocarbon as a main component. When the base oil is an ether oil (e.g., alkyl diphenyl ether) and an ester oil, dusting characteristics of the grease are unfavorably increased. Further, when the base oil having such a polar group is used, hydrolysis may occur to impair a function of the grease. In view of the above, it is preferable to avoid mixing the above oils as much as possible. The base oil preferably contains 90 mass % or more of hydrocarbon, most preferably, consists of hydrocarbon.

The hydrocarbon used as the base oil is preferably a poly-α-olefin in terms of low dusting characteristics.

A kinematic viscosity at 40 degrees C. of the base oil is preferably 400 mm²/s or less. When the kinematic viscosity at 40 degrees C. exceeds 400 mm²/s, stirring resistance is increased to excessively increase torque, thereby deteriorating wear resistance. Particularly, fretting wear may be increased. Accordingly, the kinematic viscosity at 40 degrees C. of the base oil is preferably 100 mm²/s or less, more preferably 50 mm²/s or less.

On the other hand, when the kinematic viscosity at 40 degrees C. of the base oil is less than 10 mm²/s, volatility may be increased and load resistance may be lowered.

When the kinematic viscosity at 40 degrees C. of the base oil is in a range from 10 mm²/s to 50 mm²/s, since volatility is sufficiently low and torque is reducible, the grease is advantageously used under a low load. The kinematic viscosity at 40 degrees C. of the base oil is measured in accordance with JIS K 2283.

Thickener

A thickener used for manufacturing the present grease is diurea represented by a formula (1) below.

\[ R_1, \text{NCONHR}_2, \text{NCONHR}_3 \]  

Herein, \( R_1 \) and \( R_3 \) independently represent a hydrocarbon group having 6 to 18 carbon atoms. \( R_1 \) and \( R_3 \) in diurea of the formula (1) are mutually different. Diurea of the formula (1) is generally used as a mixture. Since \( R_1 \) and \( R_3 \) are mutually different, an average ratio of a saturated hydrocarbon group in each of \( R_1 \) and \( R_3 \) is less than 100 mol %. When the average ratio of one of the above functional groups is 100 mol %, a favorable worked penetration and low dusting characteristics may not be simultaneously achieved.

Particularly, the average ratio of a cycloalkyl group in diurea is preferably in a range from 15 mol % to 85 mol % based on a total amount of an alkyl group and the cycloalkyl group (functional groups) in terms of low dusting characteristics.

Moreover, the average ratio of the cycloalkyl group is preferably in a range from 15 mol % to 50 mol % or in a range from 75 mol % to 85 mol %.

Herein, the hydrocarbon group having 6 to 18 carbon atoms is classified into a saturated one and an unsaturated one. The saturated hydrocarbon group is defined as an alkyl group and a cycloalkyl group. The unsaturated hydrocarbon group is defined as an alkenyl group. The alky group preferably has a linear structure. The alkyl group is preferably an octyl group in terms of low dusting characteristics. The cycloalkyl group is preferably a cyclohexyl group in terms of balance between the worked penetration and the low dusting characteristics. The alkenyl group is exemplified by an oleyl group.

Particularly, \( R_1 \) and \( R_2 \) are preferably an octyl group and a cyclohexyl group.

\( R_4 \) is a divalent aromatic hydrocarbon group having 6 to 13 carbon atoms. The divalent aromatic hydrocarbon group is exemplified by 4,4’-diphenylmethyl group and a tolyl group.

Diurea of the formula (1) can be easily manufactured by reacting an aromatic diisocyanate and monoamine. The aromatic diisocyanate is exemplified by diphenylmethane-4,4’-diisocyanate and tolylene-2,4’-diisocyanate. Monoamine is exemplified by octylamine and cyclohexylamine.

The Present Grease

The grease according to the exemplary embodiment contains the above base oil and the above thickener. The worked penetration of the grease according to the exemplary embodiment is in a range from 175 to 295, preferably from 180 to 275, more preferably from 180 to 265, further preferably from 230 to 265.

Herein, a content of the thickener needs to be in a range from 15 mass % to 25 mass % based on the total amount of the composition, more preferably from 15 mass % to 20 mass %. When the content of the thickener is small, the worked penetration of the grease becomes excessively high, thereby increasing the amount of the generated dust. On the other hand, when the content of the thickener is excessively large, the worked penetration of the grease becomes low, resulting in a hard grease. In this case, although the amount of the generated dust is small, penetration (permeability) of the grease to a friction surface becomes insufficient, thereby causing a poor lubrication.

When the thickener is manufactured by mixing diisocyanate and amine at the same equivalent and is mixed with the base oil to manufacture the grease, the content of the thickener is equal to a content of diisocyanate and amine providing the thickener.

Since a predetermined amount of diurea having a specific structure is used as the thickener in the grease according to the exemplary embodiment, the dusting characteristics...
of the grease are extremely low. Further, since the base oil is formed of hydrocarbon, the grease also exhibits an excellent hydrolysis resistance. Particularly, although the worked penetration of the grease is in an easily applicable range of 230 or more to show a soft grease, the grease also exhibits low dusting characteristics. Moreover, since not necessarily containing halogen and a metal salt, the grease is suitable for a precision electronic device.

Softening Treatment of The Present Grease

By stirring the present grease by centrifugal force, the worked penetration of the present grease is increased, but the dusting characteristics thereof are decreased. Generally, when the consistency of the grease is increased, the dusting characteristics are increased. Accordingly, the grease having the above performance is noticeable.

The centrifugal force is preferably 200 G or more, more preferably 300 G or more.

In the present grease according to the exemplary embodiment, additives such as an antioxidant, rust inhibitor, solid lubricant, filler, oiliness agent, and metal deactivator may be added as needed within a range where an object of the invention is achieved.

Examples of the antioxidant include: an amine antioxidant such as alkylated diphenylamine, phenyl-α-naphthylamine and alkylated-α-naphthylamine; and a phenol antioxidant such as 2,6-di-t-butyl-4-methylphenol and 4,4’-methylenebis(2,6-di-t-butylphenol). These antioxidants are used in a ratio of the order of 0.05 mass % to 2 mass %.

Examples of the rust inhibitor include: sodium nitrite, petroleum sulphonate, sorbitan monooleate, fatty acid soap and an amine compound.

Examples of the solid lubricant include polynimide, PTFE, graphite, metal oxide, boron nitride, melamine cyanurate (MCA) and molybdenum disulphide.

The above various additives may be contained singly or in combination of several kinds thereof.

EXAMPLES

Next, the invention will be further described in detail with Examples and Comparatives, which by no means limit the scope of the invention.

Specifically, various greases were manufactured by a later-described method and properties of the greases were evaluated by comparison. Each of the properties of the greases was obtained in accordance with methods described below.

(1) Worked Penetration of Grease

A worked penetration was measured in accordance with JIS K 2220.7.

(2) Amount of Generated Dust (Dust Generation Test)

Using a ball screw provided in a clean room of the class 2 defined in accordance with ISO 14644-1, a degree of dust generation from each of the greases was evaluated. Specifically, a 10-g grease was filled entirely over a screw surface of a ball screw (diameter: 16 mm, lead: 8 mm). A 50-hour operation was carried out under the conditions of a ball-nut speed being 100 mm/s and a stroke being 150 mm. Air was sucked from an intake port (sucking speed: 31 L/min) provided immediately near the screw at a middle of reciprocation. Fine particles of 0.3 μm or more were counted by a particle counter (manufactured by RION CO., LTD.: KC-03B) and defined as a dust generation number. A total counted number during the test time (50 hours) was shown in piece(s) per 10L and evaluated based on the following standard.

- A: 0 piece/10 L to 500 pieces/10 L
- A: 501 pieces/10 L to 1500 pieces/10 L
- A: 1501 pieces/10 L or more

Example 1

A grease was manufactured as follows. It should be noted that the poly-α-olefin used as the base oil was also used in other Examples and Comparatives. Properties of the poly-α-olefin were as follows.

- Kinematic Viscosity at 40 Degrees C: 46 mm²/s
- Kinematic Viscosity at 100 Degrees C: 7.7 mm²/s
- VI: 137
- Density at 15 Degrees C: 0.83 g/cm³

Manufacturing of Grease

In a separate flask, 100 g of a poly-α-olefin (the base oil) and 24.8 g of diphenylmethane-4,4’-diisocyanate (MDI: COSMONATE PH manufactured by Mitsui Chemicals, Inc.) were put. After a stirring fin was inserted, the obtained mixture was heated with stirring at 60 degrees C. at 250 rpm under a nitrogen stream. After MDI was dissolved (about 15 minutes later), a mixed amine (4.96 g of octylamine and 15.3 g of cyclohexylamine) dissolved in 50 g of the base oil was slowly added to the above resultant solution. The resultant solution was vigorously stirred once so as to entirely flow, whereby the solution was uniformed. A container of the mixed amine was washed with 5.0 g of the base oil. The used base oil was added to the separate flask. The obtained solution in the separate flask was vigorously stirred again.

Next, after the resultant solution was heated to 160 degrees C. and reached the maximum temperature, the maximum temperature was kept for one hour (while the maximum temperature was kept, the solution was vigorously stirred about once every 15 minutes, whereby the entire solution was uniformed). Subsequently, the resultant solution was cooled from being heated and was gradually cooled. After the resultant solution was cooled down to the room temperature, a finish treatment was conducted twice to the obtained product using a three-roll mill, so that grease was obtained.

Softening Treatment of Grease

The grease obtained by the above method was stirred under 400 G of centrifugal force using a rotation-revolution stirrer (MAZERUSTAR manufactured by KURAIBO INDUSTRIES LTD.) until the consistency of the grease became 2.5 to 2. A composition and evaluation results of the grease are shown in Table 1.

Example 2

A grease of Example 2 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 18.9 g of MDI, 11.3 g of octyl amine, and 5.80 g of cyclohexylamine. Then, the softening treatment was conducted to the grease of Example 2 under the same conditions. A composition and evaluation results of the grease are shown in Table 1.
Example 3

A grease of Example 3 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 27.7 g of MDI, 22.3 g of octyl amine, and 4.01 g of cyclohexylamine. Then, the softening treatment was conducted to the grease of Example 3 under the same conditions. A composition and evaluation results of the grease are shown in Table 1.

Example 4

A grease of Example 4 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 27.7 g of MDI, 22.3 g of octyl amine, and 4.01 g of cyclohexylamine and the rotation speed of the stirring flax during the manufacturing of the grease was changed from 250 rpm to 400 rpm. However, the softening treatment was not applied to the obtained grease. A composition and evaluation results of the grease are shown in Table 1.

Example 5

A grease of Example 5 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 27.7 g of MDI, 22.3 g of octyl amine, and 4.01 g of cyclohexylamine. However, the softening treatment was not conducted to the obtained grease. A composition and evaluation results of the grease are shown in Table 1.

Table 1

<table>
<thead>
<tr>
<th></th>
<th>Example 1</th>
<th>Example 2</th>
<th>Example 3</th>
<th>Example 4</th>
<th>Example 5</th>
<th>Comparative 1</th>
<th>Comparative 2</th>
<th>Comparative 3</th>
<th>Comparative 4</th>
<th>Reference Ex. 5</th>
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Comparative 1

A grease of Comparative 1 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 21.3 g of MDI and 21.3 g of octyl amine and cyclohexylamine was not used. Then, the softening treatment was conducted to the grease of Comparative 1 under the same conditions. A composition and evaluation results of the grease are shown in Table 1.

Comparative 2

A grease of Comparative 2 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 30.5 g of MDI and 23.4 g of cyclohexylamine and octyl amine was not used. Then, the softening treatment was conducted to the grease of Comparative 2 under the same conditions. A composition and evaluation results of the grease are shown in Table 1.

Comparative 3

A grease of Comparative 3 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 12.3 g of MDI, 9.84 g of n-octyl amine, and 1.89 g of cyclohexylamine. However, the softening treatment was not conducted to the obtained grease. A composition and evaluation results of the grease are shown in Table 1.

Comparative 4

A grease of Comparative 4 was manufactured in the same manner as in Example 1 except that the amounts of the reagents to be used were changed to 11.6 g of MDI and 24.4 g of n-octadecyl amine and cyclohexylamine and octylamine were not used. However, the softening treatment was not conducted to the obtained grease. A composition and evaluation results of the grease are shown in Table 1.

Reference Example

Evaluation results of a commercially available low dusting grease (AFE-CA manufactured by THK CO., LTD) are shown in Table 1.

EVALUATION RESULTS

It is found that each of the greases of Examples 1 to 5 exhibit extremely low dusting characteristics as compared with the greases of Comparatives 1 to 4 and Reference Example (commercially available grease). On the other hand, in Comparatives 1 and 3, due to the small content of the thickener for manufacturing the grease, even though a value of the worked penetration is favorable, the amount of the generated dust is large. In Comparative 2, since the worked penetration exceeds the range defined by the invention, the amount of the generated dust is large. In Comparative 4, since the amount of the thickener is small, the amount of the generated dust is slightly large although the value of the worked penetration is favorable.

As shown in Examples 4 to 5, even the aftertreatment (softening treatment) by MAZERUSTAR is not performed, the amount of the generated dust is sufficiently low.
1. A grease comprising:
a base oil comprising hydrocarbon; and
a diurea of formula (1) as a thickener in a range from 15
mass % to 25 mass % based on a total amount of the
grease, the grease exhibiting a worked penetration in a
range from 170 to 295,

\[
R_1\text{NHCONH}_2\text{NHCONH}_2R_3
\]  

(1)
wherein \(R_1\) and \(R_3\) are mutually different and are each independently a hydrocarbon group having 6 to 18 carbon atoms; and \(R_2\) is a divalent aromatic hydrocarbon group having 6 to 13 carbon atoms.

2. The grease according to claim 1, wherein the worked penetration is from 230 to 265.

3. The grease according to claim 1, wherein the hydrocarbon group is a saturated hydrocarbon group.

4. The grease according to claim 3, wherein the saturated hydrocarbon group is an alkyl group or a cycloalkyl group.

5. The grease according to claim 4, wherein the cycloalkyl group is a cyclohexyl group.

6. The grease according to claim 4, wherein \(R_1\) and \(R_3\) are each independently an octyl group or a cyclohexyl group.

7. The grease according to claim 4, wherein an average ratio of the cycloalkyl group in the diurea is from 15 mol % to 85 mol % based on a total amount of the alkyl group and the cycloalkyl group as functional groups.

8. The grease according to claim 7, wherein the average ratio of the cycloalkyl group is from 15 mol % to 50 mol %.

9. The grease according to claim 7, wherein the average ratio of the cycloalkyl group is from 75 mol % to 85 mol %.

10. The grease according to claim 1, wherein a content of the hydrocarbon is 90 mass % or more based on a total amount of the base oil.

11. The grease according to claim 1, wherein the hydrocarbon is a poly-\(\omega\)-olefin.

12. The grease according to claim 1, wherein a kinematic viscosity at 40 degrees C. of the base oil is 400 mm²/s or less.

13. A softening method of the grease according to claim 1, comprising: stirring the grease while applying centrifugal force on the grease.

* * * *