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(54) **GREASE COMPOSITION**

(57) A grease composition containing a base oil (A), a urea-based thickener (B), an antioxidant (C), and a rust inhibitor (D), wherein in a particle size distribution curve obtained by measuring the particle size of particles containing the urea-based thickener (B) in the grease composition on a volume basis with light scattering, a peak indicating the maximum frequency satisfies the following Requirements (I) and (II):

(I) a particle size indicating the maximum frequency of the peak is 1.0 μm or less; and

(II) a half width of the peak is 1.0 μm or less.

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Description

Technical Field

5 [0001] The present invention relates to a grease composition.

Background Art

10 [0002] Grease is widely used for lubricating various sliding portions of automobiles and various industrial machines because the grease easily performs sealing as compared with a lubricating oil and enables machines to have smaller size and lighter weight.

[0003] The grease is mainly composed of a base oil and a thickener. The solid nature of the grease is imparted by the thickener, and the performance of the grease varies greatly depending on the thickener used.

15 [0004] For example, urea-based grease using a urea-based thickener has a long lubricating life at a high temperature and is excellent in oxidation stability, heat resistance, and water resistance.

[0005] In addition, grease is often one mixed with various additives, together with a base oil and a thickener, for the purpose of improving desired characteristics.

20 [0006] For example, PTL 1 discloses, as a grease composition used in a hub unit bearing for a vehicle incorporated in an automobile or a railroad, a water-resistant grease composition in which an aromatic urea as a thickener and specific three kinds of rust inhibitors are blended in a base oil composed of at least one of mineral oil and synthetic oil.

Citation List

Patent Literature

25 [0007] PTL 1: JP 2008-13624 A

Summary of Invention

Technical Problem

[0008] In the meantime, there is a demand for a urea-based grease to further improve anti-wear property and friction reduction characteristics.

35 [0009] However, according to the study of the present inventors, it has been found that when an antioxidant or a rust inhibitor is blended with the urea-based grease for the purpose of improving oxidation stability, anti-wear property and friction characteristics of the grease composition may be deteriorated.

[0010] In PTL 1, there has been no study on the problem of deterioration in anti-wear property and friction characteristics of the urea-based grease composition due to the incorporation of an antioxidant or a rust inhibitor.

40 [0011] The present invention has been made to solve the above problems, and an object of the present invention is to provide a urea grease composition which is excellent in oxidation stability, anti-wear property, and friction characteristics.

Solution to Problem

45 [0012] The present inventors focused on the particle size distribution of particles containing urea-based thickener in a grease composition containing a base oil, a urea-based thickener, an antioxidant, and a rust inhibitor.

[0013] Then, it has been found that the grease composition adjusted such that the particle size and half width at the peak indicating the maximum frequency of the particle size distribution fall within the respective predetermined ranges can solve the above problem, thereby completing the present invention.

50 [0014] That is, the present invention relates to the following [1].

[1] A grease composition containing: a base oil (A), a urea-based thickener (B), an antioxidant (C), and a rust inhibitor (D),

55 wherein in a particle size distribution curve obtained by measuring the particle size of particles containing the urea-based thickener (B) in the grease composition on a volume basis with light scattering, a peak indicating the maximum frequency satisfies the following Requirements (I) and (II):

(I) a particle size indicating the maximum frequency of the peak is 1.0 μm or less; and

(II) a half width of the peak is 1.0 μm or less.

Advantageous Effects of Invention

[0015] The grease composition of the present invention has excellent oxidation stability, anti-wear property, and friction characteristics.

Brief Description of Drawings

[0016]

Fig. 1 is a schematic cross-sectional view of a grease manufacturing apparatus which can be used in one embodiment of the present invention.

Fig. 2 is a schematic cross-sectional view of the stirring portion of the grease manufacturing apparatus of Fig. 1 in the horizontal direction.

Fig. 3 is a schematic cross-sectional view of a grease manufacturing apparatus used in Comparative Example 1.

Fig. 4 is a particle size distribution curve obtained by measuring particle size of particles containing a urea-based thickener (B) in the grease composition produced in Example 1 on a volume basis with light scattering.

Fig. 5 is a particle size distribution curve obtained by measuring particle size of particles containing a urea-based thickener (B) in the grease composition produced in Comparative Example 1 on a volume basis with light scattering.

Description of Embodiments

[0017] The grease composition of the present invention contains a base oil (A), a urea-based thickener (B), an antioxidant (C), and a rust inhibitor (D). In the following description, the base oil (A), the urea-based thickener (B), the antioxidant (C), and the rust inhibitor (D) are also referred to as component (A), component (B), component (C), and component (D), respectively.

[0018] In addition, the grease composition according to one embodiment of the present invention may contain additives other than the components (A) to (D) within a range that does not impair the effects of the present invention.

[0019] In the grease composition according to one embodiment of the present invention, the total content of the components (A), (B), (C) and (D) is preferably 60 to 100% by mass, more preferably 70 to 100% by mass, still more preferably 80 to 100% by mass, and even still more preferably 90 to 100% by mass, based on the whole amount (100% by mass) of the grease composition.

[0020] According to the grease composition of the present invention, in a particle size distribution curve obtained by measuring the particle size of particles containing the urea-based thickener (B) in the grease composition on a volume basis with light scattering, a peak indicating the maximum frequency satisfies the following Requirements (I) and (II):

(I) a particle size indicating the maximum frequency of the peak is 1.0 μm or less; and

(II) a half width of the peak is 1.0 μm or less.

[0021] Requirements (I) and (II) can be said to be parameters indicating the state of aggregation of the urea thickener (B) in the grease composition blended with additives, such as the antioxidant (C) and the rust inhibitor (D), together with the base oil (A) and the urea-based thickener (B).

[0022] Here, the "particles containing the urea-based thickener (B)" as a measurement target includes not only particles formed by aggregation of the urea-based thickener (B), but also particles in which additives such as the antioxidant (C) and the rust inhibitor (D) are incorporated and aggregated together with the urea-based thickener (B).

[0023] On the other hand, aggregates which are composed only of additives such as the antioxidant (C) and the rust inhibitor (D) and which do not contain the urea-based thickener (B) are excluded from the above-mentioned "particles containing the urea-based thickener (B)". Here, "excluded" means that aggregates composed only of additives such as the antioxidant (C) and the rust inhibitor (D) are very small in comparison with the "particles containing the urea-based thickener (B)", so that they are hardly detected in the measurement of particle size with light scattering, and are negligible even if they are detected.

[0024] According to the study by the present inventors, as described above, it has been found that when the additive such as the antioxidant (C) or the rust inhibitor (D) is blended, the anti-wear property and the friction characteristics of the grease composition are deteriorated in some cases.

[0025] As a result of studying the cause of this adverse effect, the present inventors have assumed that the additive such as the antioxidant (C) or the rust inhibitor (D) is blended in the grease composition containing the urea-based thickener (B), and the urea-based thickener (B) is aggregated during the mixing process to form the micelle particles

(so-called "lump"), thereby reducing the anti-wear property and the friction characteristics.

[0026] The present inventors have focused on the peak indicating the maximum frequency in the particle size distribution curve obtained by measuring the particle size of particles containing the urea-based thickener (B) in the grease composition containing the additive such as the antioxidant (C) or the rust inhibitor (D) on a volume basis with light scattering.

[0027] In Requirement (I), it is stipulated that the particle size indicating the maximum frequency of the peak is 1.0 μm or less. The particle size is an index indicating the degree of aggregation of the urea-based thickener (B).

[0028] When the particle size is more than 1.0 μm , the aggregation of the urea-based thickener (B) is generally excessive, which may cause deterioration in anti-wear property and friction characteristics.

[0029] From the above viewpoint, the particle size indicating the maximum frequency of the peak defined in Requirement (I) is 1.0 μm or less, preferably 0.9 μm or less, more preferably 0.8 μm or less, still more preferably 0.7 μm or less, and even still more preferably 0.6 μm or less, and is usually 0.01 μm or more.

[0030] The particle size indicating the maximum frequency of the peak means the value of the particle size at the apex of the peak.

[0031] In Requirement (II), it is stipulated that the half width of the peak is 1.0 μm or less. The half width is an index indicating the distribution state of particles including urea-based thickener (B) which has a particle size larger than the particle size indicating the maximum frequency defined in Requirement (I). Here, the half width of the peak defined in Requirement (II) represents the spreading range of the particle size at 50% of the maximum frequency of Requirement (I) in the particle size distribution curve obtained by measuring the particle size of the particles on a volume basis with light scattering.

[0032] That is, when the half width is more than 1.0 μm , it can be said that the micelle particles of the urea-based thickener (B), which are excessively larger than the particle size defined in Requirement (I), are dispersed in a large amount. As a result, it is assumed that the presence of large micelle particles leads to a reduction in anti-wear property and friction characteristics.

[0033] From the above viewpoint, the half width of the peak defined in Requirement (II) is 1.0 μm or less, preferably 0.9 μm or less, more preferably 0.8 μm or less, still more preferably 0.7 μm or less, and even still more preferably 0.6 μm or less, and is usually 0.01 μm or more.

[0034] In this specification, the values defined in the above Requirements (I) and (II) are values calculated from the particle size distribution curve measured by the method of the examples described later.

[0035] Further, the values defined in Requirements (I) and (II) can be adjusted by appropriately selecting the types, properties, and contents of the components contained in the grease composition, the preparation conditions of the urea-based thickener (B), and the blending conditions of additives such as the antioxidant (C) and the rust inhibitor (D).

[0036] However, the values defined in Requirements (I) and (II) are relatively influenced by the production conditions of the urea-based thickener (B) and the blending conditions of the additives.

[0037] The details of each component contained in the grease composition of the present invention will be described below while paying attention to a specific means for adjusting the values defined in Requirements (I) and (II).

<Base oil (A)>

[0038] As the base oil (A) contained in the grease composition of the present invention, one or more kinds selected from mineral oil and synthetic oil may be used.

[0039] Examples of the mineral oil include a distillate oil obtained by atmospheric distillation or vacuum distillation of paraffin crude oil, intermediate base crude oil, or naphthenic crude oil, and a refined oil obtained by refining these distillate oils according to a conventional method.

[0040] Examples of the refining process include solvent dewaxing treatment, hydroisomerization treatment, hydrofinishing treatment, and clay treatment.

[0041] Examples of the synthetic oil include a hydrocarbon oil, an aromatic oil, an ester oil, an ether oil, a synthetic oil obtained by isomerizing a wax (GTL wax) produced by Fischer-Tropsch process, or the like.

[0042] Examples of the hydrocarbon oil include normal paraffin, iso-paraffin, polybutene, polyisobutylene, poly- α -olefin (PAO) such as 1-decene oligomer, 1-decene and ethylene co-oligomer, and the like, and a hydride thereof.

[0043] Examples of the aromatic oil include alkylbenzenes such as monoalkylbenzene and dialkylbenzene; alkylnaphthalenes such as monoalkylnaphthalene, dialkylnaphthalene, and polyalkylnaphthalene.

[0044] Examples of the ester oil include diester oils such as dibutyl sebacate, di-2-ethylhexyl sebacate, dioctyl adipate, diisodecyl adipate, ditridecyl adipate, ditridecyl glutarate, and methyl acetyl ricinoleate; aromatic ester oils such as trioctyl trimellitate, tridecyl trimellitate, and tetraoctyl pyromellitate; polyol ester oils such as trimethylolpropane caprylate, trimethylolpropane pelargonate, pentaerythritol-2-ethylhexanoate, and pentaerythritol pelargonate; and complex ester oils such as oligoester of a polyhydric alcohol and a mixed fatty acid of a dibasic acid and a monobasic acid.

[0045] Examples of the ether oil include polyglycols such as polyethylene glycol, polypropylene glycol, polyethylene glycol monoether, and polypropylene glycol monoether; phenyl ether oils such as monoalkyl triphenyl ether, alkyl diphenyl

ether, dialkyl diphenyl ether, pentaphenyl ether, tetraphenyl ether, monoalkyl tetraphenyl ether, and dialkyl tetraphenyl ether.

[0046] The kinematic viscosity at 40°C of the base oil (A) used in one embodiment of the present invention is preferably 10 to 130 mm²/s, more preferably 15 to 110 mm²/s, and still more preferably 20 to 100 mm²/s.

[0047] The base oil (A) used in one embodiment of the present invention may be a mixed base oil having a kinematic viscosity adjusted within the above range by combining a base oil having a high viscosity and a base oil having a low viscosity.

[0048] The viscosity index of the base oil (A) used in one embodiment of the present invention is preferably 60 or more, more preferably 70 or more, and still more preferably 80 or more.

[0049] In this specification, the kinematic viscosity and the viscosity index mean values measured in accordance with JIS K2283:2003.

[0050] In the grease composition according to one embodiment of the present invention, the content of the base oil (A) is preferably 50% by mass or more, more preferably 55% by mass or more, still more preferably 60% by mass or more, and even still more preferably 65% by mass or more, and preferably 98.5% by mass or less, more preferably 97% by mass or less, still more preferably 95% by mass or less, and even still more preferably 93% by mass or less, based on the whole amount (100% by mass) of the grease composition.

<Urea-based thickener (B)>

[0051] The urea-based thickener (B) contained in the grease composition of the present invention may be a compound having a urea bond, but is preferably a diurea having two urea bonds, and more preferably a compound represented by the following general formula (b1).



[0052] The urea-based thickener (B) used in one embodiment of the present invention may be composed of one type or a mixture of two or more types.

[0053] In the general formula (b1), R¹ and R² each independently represent a monovalent hydrocarbon group having 6 to 24 carbon atoms, and R¹ and R² may be the same as or different from each other. R³ represents a divalent aromatic hydrocarbon group having 6 to 18 carbon atoms.

[0054] The carbon number of the monovalent hydrocarbon group that can be selected as R¹ and R² in the general formula (b1) is 6 to 24, but it is preferably 6 to 20, and more preferably 6 to 18.

[0055] Examples of the monovalent hydrocarbon groups that can be selected as R¹ and R² include saturated or unsaturated monovalent chain hydrocarbon groups, saturated or unsaturated monovalent alicyclic hydrocarbon groups, and monovalent aromatic hydrocarbon groups, and preferably saturated or unsaturated monovalent chain hydrocarbon groups.

[0056] Examples of monovalent saturated chain hydrocarbon groups include straight-chain or branched-chain alkyl groups having 6 to 24 carbon atoms, and specific examples include a hexyl group, a heptyl group, an octyl group, a nonyl group, a decyl group, an undecyl group, a dodecyl group, a tridecyl group, a tetradecyl group, a pentadecyl group, a hexadecyl group, a heptadecyl group, an octadecyl group, an octadecenyl group, a nonadecyl group, and an icosyl group.

[0057] Examples of monovalent unsaturated chain hydrocarbon groups include straight-chain or branched-chain alkenyl group having 6 to 24 carbon atoms, and specific examples include a hexenyl group, a heptenyl group, an octenyl group, a nonenyl group, a decenyl group, a dodecenyl group, a tridecenyl group, a tetradecenyl group, a pentadecenyl group, a hexadecenyl group, an octadecenyl group, a nonadecenyl group, an icosenyl group, an oleyl group, a geranyl group, a farnesyl group, and a linoleyl group.

[0058] The monovalent saturated chain hydrocarbon group and monovalent unsaturated chain hydrocarbon group may be a straight chain group or a branched chain group.

[0059] Examples of monovalent saturated alicyclic hydrocarbon groups include cycloalkyl groups, such as a cyclohexyl group, a cycloheptyl group, a cyclooctyl group, and a cyclononyl group; and cycloalkyl groups substituted with an alkyl group having 1 to 6 carbon atoms (preferably cyclohexyl groups substituted with an alkyl group having 1 to 6 carbon atoms), such as a methylcyclohexyl group, a dimethylcyclohexyl group, an ethylcyclohexyl group, a diethylcyclohexyl group, a propylcyclohexyl group, an isopropylcyclohexyl group, a 1-methyl-propylcyclohexyl group, a butylcyclohexyl group, a pentylcyclohexyl group, a pentyl-methylcyclohexyl group, and a hexylcyclohexyl group.

[0060] Examples of monovalent unsaturated alicyclic hydrocarbon groups include cycloalkenyl groups, such as a cyclohexenyl group, a cycloheptenyl group, and a cyclooctenyl group; and cycloalkenyl groups substituted with an alkyl group having 1 to 6 carbon atoms (preferably cyclohexenyl groups substituted with an alkyl group having 1 to 6 carbon atoms), such as a methylcyclohexenyl group, a dimethylcyclohexenyl group, an ethylcyclohexenyl group, a diethylcy-

clohexenyl group, and a propylcyclohexenyl group.

[0061] Examples of monovalent aromatic hydrocarbon groups include a phenyl group, a biphenyl group, a terphenyl group, a naphthyl group, a diphenylmethyl group, a diphenylethyl group, a diphenylpropyl group, a methylphenyl group, a dimethylphenyl group, an ethylphenyl group, and a propylphenyl group.

[0062] The carbon number of the divalent aromatic hydrocarbon group that can be selected as R^3 in the general formula (b1) is 6 to 18, but it is preferably 6 to 15, and more preferably 6 to 13.

[0063] Examples of the divalent aromatic hydrocarbon group that can be selected as R^3 include a phenylene group, a diphenylmethylene group, a diphenylethylene group, a diphenylpropylene group, a methylphenylene group, a dimethylphenylene group, and an ethylphenylene group.

[0064] Among them, a phenylene group, a diphenylmethylene group, a diphenylethylene group, or a diphenylpropylene group is preferred, and a diphenylmethylene group is more preferred.

[0065] In the grease composition according to one embodiment of the present invention, the content of component (B) is preferably 1 to 40% by mass, more preferably 2 to 30% by mass, still more preferably 4 to 25% by mass, and even still more preferably 6 to 20% by mass, based on the whole amount (100% by mass) of the grease composition.

[0066] When the content of component (B) is 1% by mass or more, it is easy to adjust the worked penetration of the resulting grease composition to a suitable range.

[0067] On the other hand, when the content of component (B) is 40% by mass or less, the resulting grease composition does not become too hard, and it is possible to suppress an adverse effect such as seizure of a portion to be lubricated, such as a bearing, a sliding portion, or a joint portion of the device, to the member, which may be caused by poor lubrication.

<Method for Producing Urea-based Thickener (B)>

[0068] The urea-based thickener (B) can be usually obtained by reacting an isocyanate compound with a monoamine. The reaction is preferably performed by adding a solution β obtained by dissolving a monoamine in the base oil (A) to a heated solution α obtained by dissolving the isocyanate compound in the base oil (A).

[0069] For example, when a compound represented by the general formula (b1) is synthesized, a diisocyanate having a group corresponding to a divalent aromatic hydrocarbon group represented by R^3 in the general formula (b1) is used as an isocyanate compound, and an amine having a group corresponding to a monovalent hydrocarbon group represented by R^1 and R^2 is used as a monoamine, thus synthesizing a desired urea-based thickener (B) according to the above-described method.

[0070] In order to satisfy Requirements (I) and (II), from the viewpoint of dispersing the urea-based thickener (B) in the grease composition, it is preferable to produce component (B) by using a grease manufacturing apparatus as shown in the following [1].

[1] A grease manufacturing apparatus including:

a container body having an introduction portion into which a grease raw material is introduced and a discharge portion for discharging grease to the outside; and

a rotor having a rotation axis in an axial direction of an inner periphery of the container body and rotatably provided inside the container body,

wherein the rotor has a first concave-convex portion (i) in which concave and convex are alternately provided along the surface of the rotor, and the concave and convex are inclined with respect to the rotation axis, and (ii) which has feeding ability in the direction from the introduction portion to the discharge portion direction.

[0071] The grease manufacturing apparatus described in [1] above will be described below, but the "preferred" provisions of the following description will be made in view of dispersing the urea-based thickener (B) in the grease composition so as to satisfy Requirements (I) and (II) unless otherwise specified.

[0072] Fig. 1 is a schematic cross-sectional view of the grease manufacturing apparatus according to [1] that can be used in one embodiment of the present invention.

[0073] A grease manufacturing apparatus 1 shown in Fig. 1 includes a container body 2 for introducing a grease raw material into the inside thereof, and a rotor 3 having a rotation axis 12 on a central axis line of an inner periphery of the container body 2 and rotating around the rotation axis 12 as a center axis.

[0074] The rotor 3 rotates at high speed around the rotation axis 12 as a center axis to apply a high shearing force to a grease raw material inside the container body 2. Thus, the grease containing the urea-based thickener (B) is produced.

[0075] As shown in Fig. 1, the container body 2 is preferably partitioned from an upper portion to an introduction portion 4, a retention portion 5, a first inner peripheral surface 6, a second inner peripheral surface 7, and a discharge portion 8.

[0076] As shown in Fig. 1, it is preferable that the container body 2 has an inner peripheral surface forming such a truncated cone shape that an inner diameter thereof gradually increases from the introduction portion 4 toward the

discharge portion 8.

[0077] The introduction portion 4 serving as one end of the container body 2 is provided with a plurality of solution introducing pipes 4A and 4B for introducing a grease raw material from the outside of the container body 2.

[0078] The retention portion 5 is disposed in a lower portion of the introduction portion 4, and is a space for temporarily retaining the grease raw material introduced from the introduction portion 4. When the grease raw material is retained for a long time in the retention portion 5, grease adhering to the inner peripheral surface of the retention portion 5 forms a large lump, so that it is preferable to transport the grease to the first inner peripheral surface 6 in the downstream side in a short time. Further, it is preferable to directly transport to the first inner peripheral surface 6 without passing through the retention portion 5.

[0079] The first inner peripheral surface 6 is disposed at a lower portion adjacent to the retention portion 5, and the second inner peripheral surface 7 is disposed at a lower portion adjacent to the first inner peripheral surface 6. As will be described in detail later, it is preferable to provide a first concave-convex portion 9 on the first inner peripheral surface 6 and to provide a second concave-convex portion 10 on the second inner peripheral surface 7, in order to cause the first inner peripheral surface 6 and the second inner peripheral surface 7 to function as a high shearing portion for imparting a high shearing force to the grease raw material or grease.

[0080] The discharge portion 8 serving as the other end of the container body 2 is a part for discharging grease agitated by the first inner peripheral surface 6 and the second inner peripheral surface 7, and includes a discharge port 11 for discharging grease. The discharge port 11 is formed in a horizontal direction orthogonal to the rotation axis 12. As a result, grease is discharged from the discharge port 11 in the horizontal direction.

[0081] The rotor 3 is rotatably provided on the center axis line of the peripheral surface of the container body 2, which has a truncated cone shape, as a rotation axis 12, and rotates counterclockwise when the container body 2 is viewed from the upper portion to the lower portion as shown in Fig. 1.

[0082] The rotor 3 has an outer peripheral surface that expands in accordance with the enlargement of the inner diameter of the truncated cone of the container body 2, and the outer peripheral surface of the rotor 3 and the inner peripheral surface of the truncated cone of the container body 2 are maintained at a constant interval.

[0083] On the outer peripheral surface of the rotor 3, a first concave-convex portion 13 of the rotor in which concave and convex are alternately provided along the surface of the rotor 3 is provided.

[0084] The first concave-convex portion 13 of the rotor is inclined to the rotation axis 12 of the rotor 3 from the introduction portion 4 toward the discharge portion 8, and has the feeding ability from the introduction portion 4 to the discharge portion 8. That is, the first concave-convex portion 13 of the rotor is inclined in the direction in which the solution is pushed toward the downstream side when the rotor 3 rotates in the direction shown in Fig. 1.

[0085] The step difference between a concave portion 13A and a convex portion 13B of the first concave-convex portion 13 of the rotor is preferably 0.3 to 30, more preferably 0.5 to 15, and still more preferably 2 to 7, when the diameter of the concave portion 13A on the outer peripheral surface of the rotor 3 is 100.

[0086] The number of convex portions 13B of the first concave-convex portion 13 of the rotor in the circumferential direction is preferably 2 to 1000, more preferably 6 to 500, and still more preferably 12 to 200.

[0087] The ratio of the width of the convex portion 13B to the width of the concave portion 13A of the first concave-convex portion 13 of the rotor [the width of the convex portion/the width of the concave portion] in the cross section orthogonal to the rotation axis 12 of the rotor 3 is preferably 0.01 to 100, more preferably 0.1 to 10, and still more preferably 0.5 to 2.

[0088] The inclination angle of the first concave-convex portion 13 of the rotor with respect to the rotation axis 12 is preferably 2 to 85 degrees, more preferably 3 to 45 degrees, and still more preferably 5 to 20 degrees.

[0089] It is preferable that the first inner peripheral surface 6 of the container body 2 is provided with the first concave-convex portion 9 formed with a plurality of concave and convex along the inner peripheral surface thereof.

[0090] It is preferable that the concave and convex of the first concave-convex portion 9 on the container side are inclined in the opposite direction to the first concave-convex portion 13 of the rotor.

[0091] That is, it is preferable that the plurality of concave and convex of the first concave-convex portion 9 on the container side be inclined in the direction in which the solution is pushed toward the downstream side when the rotation axis 12 of the rotor 3 rotates in the direction shown in Fig. 1. The stirring capacity and the discharge capacity are further enhanced by the first concave-convex portion 9 having a plurality of concave and convex provided on the first inner peripheral surface 6 of the container body 2.

[0092] The depth of the concave and convex of the first concave-convex portion 9 on the container side is preferably 0.2 to 30, more preferably 0.5 to 15, and still more preferably 1 to 5, when the inner diameter (diameter) of the container is set to 100.

[0093] The number of concave and convex of the first concave-convex portion 9 on the container side is preferably 2 to 1000, more preferably 6 to 500, and still more preferably 12 to 200.

[0094] The ratio of the width of the concave portion to the width of the convex portion between grooves in the concave and convex of the first concave-convex portion 9 on the container side (width of the concave portion/width of the convex

portion) is preferably 0.01 to 100, more preferably 0.1 to 10, and still more preferably 0.5 to 2 or less.

[0095] The inclination angle of the concave and convex of the first concave-convex portion 9 on the container side with respect to the rotation axis 12 is preferably 2 to 85 degrees, more preferably 3 to 45 degrees, and still more preferably 5 to 20 degrees.

[0096] By providing the first concave-convex portion 9 on the first inner peripheral surface 6 of the container body, the first inner peripheral surface 6 can be made to function as a high shearing portion for imparting high shearing force to the grease raw material or grease, but the first concave-convex portion 9 does not necessarily have to be provided.

[0097] It is preferable that a second concave-convex portion 14 of a rotor having concave and convex alternately provided along the surface of the rotor 3 is provided on the outer peripheral surface of the lower part of the first concave-convex portion 13 of the rotor.

[0098] The second concave-convex portion 14 of the rotor is inclined with respect to the rotation axis 12 of the rotor 3, and has a feeding suppression ability to push the solution back toward the upstream side from the introduction portion 4 toward the discharge portion 8.

[0099] The step of the second concave-convex portion 14 of the rotor is preferably 0.3 to 30, more preferably 0.5 to 15, and still more preferably 2 to 7, when the diameter of the concave portion of the outer peripheral surface of the rotor 3 is set to 100.

[0100] The number of convex portions of the second concave-convex portion 14 of the rotor in the circumferential direction is preferably 2 to 1000, more preferably 6 to 500, and still more preferably 12 to 200.

[0101] The ratio of the width of the convex portion to the width of the concave portion of the second concave-convex portion 14 of the rotor in a cross section orthogonal to the rotation axis of the rotor 3 (the width of the convex portion/the width of the concave portion) is preferably 0.01 to 100, more preferably 0.1 to 10, and still more preferably 0.5 to 2.

[0102] The inclination angle of the second concave-convex portion 14 of the rotor with respect to the rotation axis 12 is preferably 2 to 85 degrees, more preferably 3 to 45 degrees, and still more preferably 5 to 20 degrees.

[0103] It is preferable that the second inner peripheral surface 7 of the container body 2 is provided with the second concave-convex portion 10 formed with a plurality of concave and convex adjacent to the lower portion of the concave and convex in the first concave-convex portion 9 on the container side.

[0104] It is preferable that the plurality of concave and convex of the second concave-convex portion 10 on the container side are formed on the inner peripheral surface of the container body 2, and that the concave and convex are inclined in opposite directions to the inclination direction of the second concave-convex portion 14 of the rotor.

[0105] That is, it is preferable that the plurality of concave and convex of the second concave-convex portion 10 on the container side are inclined in the direction in which the solution is pushed back toward the upstream side when the rotation axis 12 of the rotor 3 rotates in the direction shown in Fig. 1. The stirring capacity is further enhanced by the concave and convex of the second concave-convex portion 10 provided on the second inner peripheral surface 7 of the container body 2. Further, the second inner peripheral surface 7 of the container body can function as a high shearing portion which imparts a high shearing force to the grease raw material or grease.

[0106] The depth of the concave portion of the second concave-convex portion 10 on the container side is preferably 0.2 to 30, more preferably 0.5 to 15, and still more preferably 1 to 5, when the inner diameter (diameter) of the container is set to 100.

[0107] The number of concave portions of the second concave-convex portion 10 on the container side is preferably 2 to 1000, more preferably 6 to 500, and still more preferably 12 to 200.

[0108] The ratio of the width of the convex portion of the concave and convex of the second concave-convex portion 10 on the container side to the width of the concave portion in the cross section orthogonal to the rotation axis 12 of the rotor 3 [width of the convex portion / width of the concave portion] is preferably 0.01 to 100, more preferably 0.1 to 10, and still more preferably 0.5 to 2 or less.

[0109] The inclination angle of the second concave-convex portion 10 on the container side with respect to the rotation axis 12 is preferably 2 to 85 degrees, more preferably 3 to 45 degrees, and still more preferably 5 to 20 degrees.

[0110] The ratio of the length of the first concave-convex portion 9 on the container side to the length of the second concave-convex portion 10 on the container side [length of the first concave-convex portion / length of the second concave-convex portion] is preferably 2/1 to 20/1.

[0111] Fig. 2 is a horizontal cross-sectional view of the first concave-convex portion 9 on the container side of the grease manufacturing apparatus 1.

[0112] In the first concave-convex portion 13 shown in Fig. 2, a plurality of scrapers 15 each having a tip protruding toward the inner peripheral surface side of the container body 2 are provided more than the tip end in the projecting direction of the convex portion 13B of the first concave-convex portion 13. Further, although not shown, the second concave-convex portion 14 is also provided with a plurality of scrapers in which the tip of the convex portion protrudes toward the inner peripheral surface side of the container body 2, similarly to the first concave-convex portion 13.

[0113] The scraper 15 scrapes off the grease adhering to the inner peripheral surface of the first concave-convex portion 9 on the container side and the second concave-convex portion 10 on the container side.

[0114] With respect to the protrusion amount of the tip end of the scraper 15 relative to the projecting amount of the projecting portion 13 B of the first concave-convex portion 13 of the rotor, the ratio $[R2/R1]$ of the radius (R2) of the tip of the scraper 15 to the radius (R1) of the tip of the convex portion 13B is preferably more than 1.005 and less than 2.0.

[0115] The number of scrapers 15 is preferably 2 to 500, more preferably 2 to 50, and still more preferably 2 to 10.

[0116] In the grease manufacturing apparatus 1 shown in Fig. 2, the scraper 15 is provided, but may not be provided, or may be provided intermittently.

[0117] In order to produce the grease containing the urea-based thickener (B) by the grease manufacturing apparatus 1, the solution α and the solution β which are the aforementioned grease raw materials are introduced respectively from the solution introducing pipes 4A and 4B of the introduction portion 4 of the container body 2, and the rotor 3 is rotated at high speed to produce the grease containing the urea-based thickener (B).

[0118] Then, by using the obtained grease, the urea-based thickener (B) can be dispersed in the grease composition so as to satisfy the above Requirements (I) and (II) even if an additive containing an antioxidant (C) and a rust inhibitor (D) is blended.

[0119] As a high-speed rotation condition of the rotor 3, the shear rate applied to the grease raw material is preferably 10^2 s^{-1} or more, more preferably 10^3 s^{-1} or more, still more preferably 10^4 s^{-1} or more, and is usually 10^7 s^{-1} or less.

[0120] In addition, the ratio of the maximum shear rate (Max) to the minimum shear rate (Min) in the shearing at the time of high-speed rotation of the rotor 3 (Max/Min) is preferably 100 or less, more preferably 50 or less, and still more preferably 10 or less.

[0121] Since the shear rate to the mixed solution is as uniform as possible, the dispersion state of the thickener and its precursor is improved and a uniform grease structure is obtained.

[0122] Here, the maximum shear rate (Max) is the highest shear rate applied to the mixed solution, and the minimum shear rate (Min) is the lowest shear rate applied to the mixed solution, which are defined as follows.

Maximum shear rate (Max) = (linear velocity at the tip of the convex portion 13B of the first concave-convex portion 13 of the rotor) / (a gap A1 between the tip end of the convex portion 13B of the first concave-convex portion 13 of the rotor and the convex portion of the first concave-convex portion 9 on the container side of the first concave-convex portion 6).

Minimum shear rate (Min) = (linear velocity of the concave portion 13A of the first concave-convex portion 13 of the rotor) / (a gap A2 between the concave portion 13A of the first concave-convex portion 13 of the rotor and the concave portion of the first concave-convex portion 9 on the container side of the first concave-convex portion 6).

[0123] The gap A1 and the gap A2 are as shown in Fig. 2.

[0124] Since the grease manufacturing apparatus 1 is provided with the scraper 15, grease adhering to the inner peripheral surface of the container body 2 can be scraped off, so that the generation of the lumps during kneading can be prevented, and the grease in which the urea-based thickener (B) is highly dispersed can be continuously produced in a short time.

[0125] Moreover, since the scraper 15 scrapes off the grease adhered thereto, it is possible to prevent the retained grease from becoming a resistance to rotation of the rotor 3, so that the rotational torque of the rotor 3 can be reduced, and the power consumption of the drive source can be reduced, thereby making it possible to continuously manufacture the grease efficiently.

[0126] Since the inner peripheral surface of the container body 2 is in a shape of a truncated cone whose inner diameter increases from the introduction portion 4 toward the discharge portion 8, the centrifugal force has an effect of discharging the grease or grease raw material in the downstream direction, and the rotation torque of the rotor 3 can be reduced to continuously manufacture the grease.

[0127] Since the first concave-convex portion 13 of the rotor is provided on an outer peripheral surface of the rotor 3, the first concave-convex portion 13 of the rotor is inclined with respect to the rotation axis 12 of the rotor 3, and a feeding

ability from the introduction portion 4 to the discharge portion 8 is provided, and the second concave-convex portion 14 of the rotor is inclined with respect to the rotation axis 12 of the rotor 3, and a feeding suppression ability from the introduction portion 4 to the discharge portion 8 is provided, a high shear force can be applied to the solution, and the urea-based thickener (B) can be dispersed in the grease composition so as to satisfy the above-mentioned Requirements (I) and (II) even after blending the additive.

[0128] Since the first concave-convex portion 9 on the container side is formed on the first inner peripheral surface 6 of the container body and is inclined in the opposite direction to the first concave-convex portion 13 of the rotor, in addition to the effect of the first concave-convex portion 13 of the rotor, sufficient stirring of grease raw material can be carried out while extruding grease or grease raw material in the downstream direction, and the urea-based thickener (B) can be dispersed in the grease composition so as to satisfy the above Requirements (I) and (II) even after blending the additive.

[0129] Further, since the second concave-convex portion 10 on the container side is provided on the second inner peripheral surface 7 of the container body and the second concave-convex portion 14 of the rotor is provided on the outer peripheral surface of the rotor 3, the grease raw material can be prevented from flowing out from the first inner peripheral surface 6 of the container body more than necessary, so that the urea-based thickener (B) can be dispersed in the grease composition so as to satisfy Requirements (I) and (II) even after blending the additive by giving high shear force to the solution to highly disperse the grease raw material.

<Antioxidant (C)>

[0130] The antioxidant (C) contained in the grease composition of the present invention may be a compound capable of imparting antioxidant performance, and preferably contains one or more kinds selected from an amine antioxidant (C1) and a phenol-based antioxidant (C2).

[0131] The antioxidant (C) used in one embodiment of the present invention may be used alone or in combination of two or more thereof.

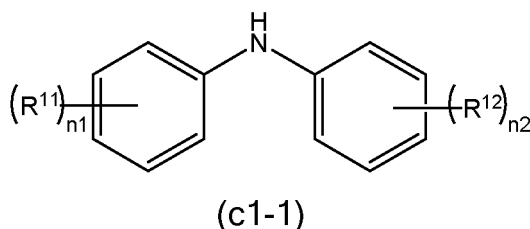
[0132] As the amine antioxidant (C1), a compound having an amino group may be used, and a diphenylamine compound and a naphthylamine compound are preferred.

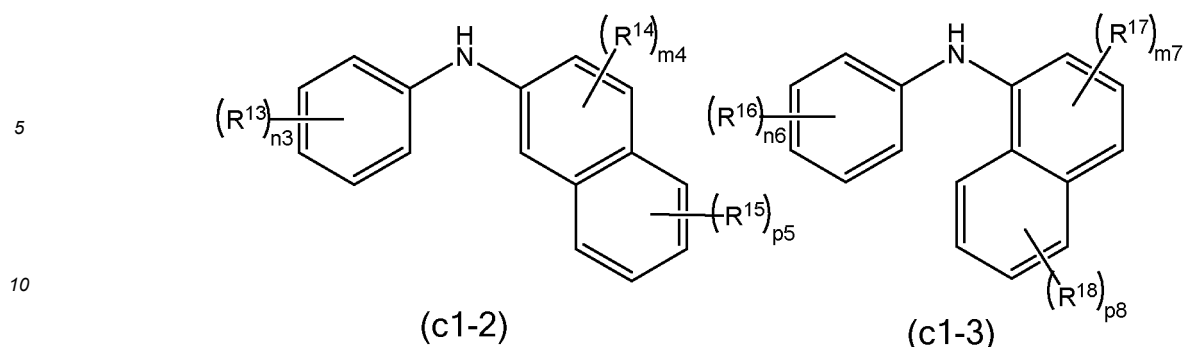
[0133] Examples of the diphenylamine compound include monoalkyldiphenylamine compounds having one alkyl group having 1 to 30 carbon atoms (preferably 4 to 30, more preferably 8 to 30), such as mono-octyl diphenylamine, and monononyl diphenylamine; dialkyldiphenylamine compounds having two alkyl groups having 1 to 30 carbon atoms (preferably 4 to 30, more preferably 8 to 30), such as 4,4'-dibutyldiphenylamine, 4,4'-dipentyldiphenylamine, 4,4'-dihexyldiphenylamine, 4,4'-diheptyldiphenylamine, 4,4'-dioctyldiphenylamine, and 4,4'-dinonyldiphenylamine; polyalkyldiphenylamine compounds having 3 or more alkyl groups having 1 to 30 carbon atoms (preferably 4 to 30 and more preferably 8 to 30), such as tetrabutyldiphenylamine, tetrahexyldiphenylamine, tetraoctyldiphenylamine, and tetranonyldiphenylamine; and 4,4'-bis (α,α -dimethylbenzyl)diphenylamine.

[0134] Examples of the naphthylamine compound include 1-naphthylamine, phenyl-1-naphthylamine, butylphenyl-1-naphthylamine, pentylphenyl-1-naphthylamine, hexylphenyl-1-naphthylamine, heptylphenyl-1-naphthylamine, octylphenyl-1-naphthylamine, nonylphenyl-1-naphthylamine, decylphenyl-1-naphthylamine, and dodecylphenyl-1-naphthylamine.

[0135] Among the diphenylamine compounds, a compound represented by the following general formula (c1-1) is preferable.

[0136] Among the naphthylamine compounds, a compound represented by the following general formula (c1-2) or a compound represented by the following general formula (c1-3) is preferable.





[0137] In the above general formulae (c1-1), (c1-2), and (c1-3), R^{11} to R^{18} are each independently an alkyl group having 1 to 20 carbon atoms (preferably 4 to 18, more preferably 6 to 16, and still more preferably 8 to 14). Examples of the alkyl group include the same alkyl groups as those having 1 to 20 carbon atoms among the alkyl groups that the alkylbenzene (B) may have.

n_1 , n_2 , n_3 , and n_6 are each independently an integer of 0 to 5, preferably an integer of 0 to 3, more preferably an integer of 0 to 1, and still more preferably 1.

m_4 and m_7 are each independently an integer of 0 to 3, preferably an integer of 0 to 1, and more preferably 0.

p_5 and p_8 are each independently an integer of 0 to 4, preferably an integer of 0 to 2, more preferably an integer of 0 to 1, and still more preferably 0.

[0138] Examples of the phenol-based antioxidant (C2) include monocyclic phenol compounds such as 2,6-di-tert-butyl-4-methylphenol, 2,6-di-tert-butyl-4-ethylphenol, 2,4,6-tri-tert-butylphenol, 2,6-di-tert-butyl-4-hydroxymethylphenol, 2,6-di-tert-butylphenol, 2,4-dimethyl-6-tert-butylphenol, 2,6-di-tert-butyl-4-(N,N-dimethylaminomethyl)phenol, 2,6-di-tert-amyl-4-methylphenol, and n-octadecyl-3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, and polycyclic phenol compounds such as 4,4'-methylenebis(2,6-di-tert-butylphenol), 4,4'-isopropylidenebis(2,6-di-tert-butylphenol), 2,2'-methylenebis(4-methyl-6-tert-butylphenol), 4,4'-bis(2,6-di-tert-butylphenol), 4,4'-bis(2-methyl-6-tert-butylphenol), 2,2'-methylenebis(4-ethyl-6-tert-butylphenol), and 4,4'-butylidenebis(3-methyl-6-tert-butylphenol).

[0139] These phenol-based antioxidants (D2) may be used alone or in combination of two or more.

[0140] The phenol-based antioxidant (C2) may be a compound having a phenol structure, and may be a monocyclic phenol compound or a polycyclic phenol compound.

[0141] Examples of the monocyclic phenol compound include, for example, 2,6-di-tert-butyl-4-methylphenol, 2,6-di-tert-butyl-4-ethylphenol, 2,4,6-tri-tert-butylphenol, 2,6-di-tert-butyl-4-hydroxymethylphenol, 2,6-di-tert-butylphenol, 2,4-dimethyl-6-tert-butylphenol, 2,6-di-tert-butyl-4-(N,N-dimethylaminomethyl)phenol, 2,6-di-tert-amyl-4-methylphenol, and benzenepropanoic acid 3,5-bis(1,1-dimethylethyl)-4-hydroxyalkyl ester.

[0142] Examples of the polycyclic phenol compound include, for example, 4,4'-methylenebis(2,6-di-tert-butylphenol), 4,4'-isopropylidenebis(2,6-di-tert-butylphenol), 2,2'-methylenebis(4-methyl-6-tert-butylphenol), 4,4'-bis(2,6-di-tert-butylphenol), 4,4'-bis(2-methyl-6-tert-butylphenol), 2,2'-methylenebis(4-ethyl-6-tert-butylphenol), and 4,4'-butylidenebis(3-methyl-6-tert-butylphenol).

[0143] In the grease composition according to one embodiment of the present invention, the content of the component (C) is preferably 0.01 to 15% by mass, more preferably 0.05 to 10% by mass, still more preferably 0.10 to 7% by mass, and even still more preferably 0.50 to 4% by mass based on the whole amount (100% by mass) of the grease composition.

<Rust Inhibitor (D)>

[0144] The rust inhibitor (D) contained in the grease composition of the present invention may be any compound capable of imparting rust preventive performance, and examples thereof include zinc stearate, a carboxylic acid type rust inhibitor, a succinic acid derivative, a thiadiazole and derivatives thereof, benzotriazole and derivatives thereof, sodium nitrite, petroleum sulfonate, sorbitan monooleate, fatty acid soap, and amine compounds.

[0145] These rust inhibitors (D) may be used alone or in combination of two or more thereof.

[0146] As the rust inhibitor (D) used in one embodiment of the present invention, a carboxylic acid rust inhibitor is preferable.

[0147] The carboxylic acid rust inhibitor is more preferably a succinic acid ester, and more preferably an alkenyl succinic acid polyhydric alcohol ester.

[0148] The alkenyl succinic acid polyhydric alcohol ester is an ester of an alkenyl succinic acid and a polyhydric alcohol.

[0149] The alkenyl group of alkenyl succinic acid is preferably an alkenyl group having 12 to 20 carbon atoms, and specific examples include dodecenyl, hexadecenyl, octadecenyl, and isoctadecenyl.

[0150] Examples of the polyhydric alcohol include saturated dihydric alcohols having 1 to 6 carbon atoms such as ethylene glycol, propylene glycol, butylene glycol, hexylene glycol, and structural isomers thereof; trivalent or higher saturated polyhydric alcohols such as trimethylolpropane, trimethylolbutane, glycerin, pentaerythritol, and dipentaerythritol.

[0151] In the grease composition according to one embodiment of the present invention, the content of component (D) is preferably 0.01 to 5% by mass, more preferably 0.03 to 3% by mass, still more preferably 0.05 to 2% by mass, and even still more preferably 0.10 to 1% by mass based on the whole amount (100% by mass) of the grease composition.

<Other Additives>

[0152] The grease composition according to one embodiment of the present invention may contain, besides components (A) to (D), other additives, which are those blended in general grease, within a range that does not impair the effects of the present invention.

[0153] Such additives include, for example, extreme pressure agents, viscosity improvers, solid lubricants, cleaning dispersants, corrosion inhibitors, metal deactivators, and the like.

[0154] These additives may be used alone or in combination of two or more thereof.

[0155] Examples of extreme pressure agents include thiocarbamic acids such as zinc dialkyldithiophosphate, molybdenum dialkyldithiophosphate, ashless dithiocarbamate, and zinc dithiocarbamate; sulfur compounds such as sulfurized fats and oils, sulfurized olefins, polysulfide, thiophosphoric acids, thioterpenes, and dialkylthiopropionate; phosphate esters such as tricresyl phosphate; and phosphite esters such as triphenyl phosphite.

[0156] Examples of viscosity improvers include polymethacrylate (PMA), olefin copolymer (OCP), polyalkylstyrene (PAS), and styrene-diene copolymer (SCP).

[0157] Examples of solid lubricants include polyimide, PTFE, graphite, metal oxide, boron nitride, melamine cyanurate (MCA), and molybdenum disulfide.

[0158] Examples of cleaning dispersants include ashless dispersants such as succinimide and boron succinimide.

[0159] Examples of corrosion inhibitors include benzotriazole compounds and thiazole compounds.

[0160] Examples of metal deactivators include benzotriazole compounds.

[0161] In the grease composition according to one embodiment of the present invention, the contents of the other additives are each independently usually 0 to 10% by mass, preferably 0 to 7% by mass, more preferably 0 to 5% by mass, and still more preferably 0 to 2% by mass based on the whole amount (100% by mass) of the grease composition.

[0162] In the grease composition according to one embodiment of the present invention, the total content of the additive containing components (C) and (D) is preferably 1 to 100 parts by mass, more preferably 3 to 80 parts by mass, still more preferably 5 to 60 parts by mass, and even still more preferably 10 to 40 parts by mass with respect to 100 parts by mass of the whole amount of component (B).

<Method of Blending Additives>

[0163] The grease composition of the present invention can be produced by adding an additive containing components (C) and (D) to a grease containing a base oil (A) and a urea-based thickener (B) synthesized by the method described above.

[0164] However, from the viewpoint of dispersing the urea-based thickener (B) in the grease composition so as to satisfy Requirements (I) and (II), the heating temperature of the grease containing the base oil (A) and the urea-based thickener (B) in blending the additive and in stirring after the blending is preferably 80 to 200°C, more preferably 90 to 180°C, still more preferably 100 to 160°C, and even still more preferably 110 to 140°C.

[Physical Properties of Grease Composition of the Present Invention]

[0165] The worked penetration of the grease composition according to one embodiment of the present invention at 25°C is preferably 180 to 300, more preferably 200 to 290, still more preferably 220 to 285, and even still more preferably 240 to 280.

[0166] In this specification, the worked penetration of the grease composition means a value measured at 25°C in accordance with the ASTM D217 test method.

[0167] The dropping point of the grease composition according to one embodiment of the present invention is preferably 240°C or higher, more preferably 250°C or higher, still more preferably 255°C or higher, and even still more preferably 260°C or higher.

[0168] In this specification, the dropping point of the grease composition means a value measured at 25°C in accordance with JIS K2220 8:2013.

[0169] In the grease composition according to one embodiment of the present invention, the oxidation stability value

measured in accordance with the oxidation stability test described in the examples described later is preferably 100 kPa or less, more preferably 70 kPa or less, still more preferably 50 kPa or less, and even still more preferably 25 kPa or less.

[0170] For the grease composition according to one embodiment of the present invention, the amount of wear measured in accordance with the fretting wear test described in the examples described later is preferably 20 mg or less, more preferably 15 mg or less, still more preferably 10 mg or less, and even still more preferably 5 mg or less.

[0171] For the grease composition according to one embodiment of the present invention, the friction coefficient measured in accordance with the vibration friction wear test (SRV test) described in the examples described later is preferably 0.12 or less, more preferably 0.10 or less, still more preferably 0.08 or less, and even still more preferably 0.07 or less.

[Use of Grease of the Present Invention]

[0172] The grease compositions of the present invention have excellent oxidation stability, anti-wear property, and friction characteristics.

[0173] Therefore, the grease composition of the present invention can be used for lubricating portions to be lubricated, such as a bearing portion, a sliding portion, a gear portion, a joint portion, or the like, in an apparatus which is required to have such characteristics. More specifically, it is particularly preferable to use it in a bearing portion of a hub unit, an electric power steering, a driving electric motor flywheel, a ball joint, a wheel bearing, a spline portion, a constant velocity joint, a clutch booster, a servo motor, a blade bearing, a bearing portion of a generator, or the like.

[0174] Examples of the field of the device in which the grease composition of the present invention can be suitably used include an automotive field, a field of business equipment, a field of machine tools, a field of wind turbines, and a field of construction or agricultural machinery.

[0175] Examples of the portion to be lubricated in the device for the automotive field, in which the grease composition of the present invention can be suitably used, include bearing portions in a device such as a radiator fan motor, a fan coupling, an alternator, an idler pulley, a hub unit, a water pump, a power window, a wiper, an electric power steering, a driving electric motor fly wheel, a ball joint, a wheel bearing, a spline portion, and a constant velocity joint; and bearing portions, gear portions, or sliding portions in a device such as door locks, door hinges, and clutch boosters.

[0176] Examples of the portion to be lubricated portion in the device for the field of business equipment, in which the grease composition of the present invention can be suitably used, include a fixing roll in a device such as a printer, and bearing and gear portions in a device such as a polygon motor.

[0177] Examples of the portion to be lubricated in the device for the field of machine tools, in which the grease composition of the present invention can be suitably used, include bearing portions in a reduction gear such as a spindle, a servo motor, a working robot, and the like.

[0178] Examples of the portion to be lubricated in the device for the field of wind turbines, in which the grease composition of the present invention can be suitably used, include bearing portions such as a blade bearing and a generator.

[0179] Examples of the portion to be lubricated in the device for the field of construction or agricultural machinery, in which the grease composition of the present invention can be suitably used, include bearing portions, gear portions, and sliding portions such as a ball joint and a spline part.

Examples

[0180] The present invention will now be described in more detail with reference to Examples, which are not intended to limit the scope of the present invention. The measuring methods of various physical properties are as follows.

(1) Kinematic viscosity at 40°C and 100°C and Viscosity index

[0181] The measurement and calculation were carried out in accordance with JIS K2283:2003.

(2) Worked penetration

[0182] The measurement was carried out at 25°C in accordance with the ASTM D217 method.

(3) Dropping point

[0183] The measurement was carried out in accordance with JIS K2220 8:2013.

Example 1

(1) Synthesis of urea grease

- 5 **[0184]** A solution α was prepared by adding 7.96 parts by mass of diphenylmethane-4,4'-diisocyanate (MDI) to 92.04 parts by mass of a base oil, a poly α -olefin (PAO) (kinematic viscosity at 40°C: 47 mm²/s, kinematic viscosity at 100°C: 7.8 mm²/s, viscosity index: 137) heated to 70°C.
- [0185]** Separately, a solution β was prepared by adding 2.01 parts by mass of cyclohexylamine and 10.05 parts by mass of stearylamine to 87.94 parts by mass of poly α -olefin (PAO) (kinematic viscosity at 40°C: 47 mm²/s, kinematic viscosity at 100°C: 7.8 mm²/s, viscosity index: 137) heated to 70°C.
- 10 **[0186]** Then, by using the grease manufacturing apparatus 1 shown in Fig. 1, the solution α and the solution β were introduced into the container body 2 at the same time as follows. The solution α heated to 70°C was introduced into the container body 2 at a flow rate of 150 L/h from the solution introducing pipe 4A, the solution β heated to 70°C was introduced into the container body 2 at a flow rate of 150 L/h from the solution introducing pipe 4B, and the solution α and the solution β were simultaneously and continuously introduced into the container body 2 in a state in which the rotor 3 was rotated. The rotational speed of the rotor 3 of the grease manufacturing apparatus 1 used was 8000 rpm.
- 15 **[0187]** In this case, the maximum shear rate (Max) was 10,500 s⁻¹, and the ratio between the maximum shear rate (Max) and the minimum shear rate (Min) [Max/Min] was 3.5 and stirring was carried out.
- [0188]** The urea-based thickener contained in the urea grease obtained corresponds to a compound in which R¹ and R² in the general formula (b1) are a cyclohexyl group or a stearyl group (octadecyl group), and R³ is a diphenyl methylene group.
- 20

(2) Preparation of grease composition

- 25 **[0189]** While stirring the urea grease obtained in the above (1) at 120°C, 4,4-dinonyldiphenylamine as an antioxidant and an alkenyl succinic acid polyhydric alcohol ester as a rust inhibitor were added thereto.
- [0190]** After stirring for 0.5 hours, it was cooled to 25°C by natural cooling to obtain a grease composition (i).
- [0191]** The content of each component in the grease composition (i) is as shown in Table 1.

30 Comparative Example 1

(1) Synthesis of urea grease

- [0192]** The same solutions as the solution α and the solution β prepared in Example 1 were used.
- 35 **[0193]** Using the grease manufacturing apparatus shown in Fig. 3, the solution α heated to 70°C was introduced into the container body at a flow rate of 504 L/h from the solution introducing pipe. Thereafter, the solution β heated to 70°C was introduced into the container body containing the solution α at a flow rate of 144 L/h from the solution introducing pipe. After all the solution β was introduced into the container body, the stirring blade was rotated, and the temperature was raised to 160°C while stirring was continued, and the mixture was held for 1 hour to synthesize urea grease.
- 40 **[0194]** In this case, stirring was carried out such that the maximum shear rate (Max) was 42,000 s⁻¹ and the ratio between the maximum shear rate (Max) and the minimum shear rate (Min) [Max/Min] was 1.03.

(2) Preparation of grease composition

- 45 **[0195]** While stirring the urea grease obtained in the above (1) at 120°C, dinonyldiphenylamine as an antioxidant and alkenyl succinic acid polyhydric alcohol ester as a rust inhibitor were added thereto.
- [0196]** After stirring for 0.5 hours, it was cooled to 25°C by natural cooling to obtain a grease composition (ii).
- [0197]** The content of each component in the grease composition (ii) is as shown in Table 1.
- [0198]** The grease compositions prepared in Example and Comparative Example were measured for worked penetration and dropping point, and the following measurements were carried out. The results are shown in Table 1.
- 50

[Particle size distribution of particles containing urea-based thickener]

- [0199]** The prepared grease composition was vacuum-defoamed, filled in a 1 mL syringe, and a grease composition of 0.10 to 0.15 mL was extruded from the syringe, and the extruded grease composition was placed on the surface of a plate-shaped cell of a fixing jig for a paste cell.
- 55 **[0200]** Further, another plate-shaped cell was stacked on the grease composition to obtain a measurement cell in which the grease composition was sandwiched between two cells.

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[0201] Using a laser diffraction type particle size distribution analyzer (trade name: LA-920, manufactured by Horiba, Ltd.), a particle size distribution curve, on a volume basis, of the particles containing urea-based thickener contained in the grease composition in the cell for measurement was obtained.

[0202] In this particle size distribution curve, a peak having the maximum frequency was specified, and the value of the particle size indicating the maximum frequency of the peak defined by the above Requirement (I) and the half width of the peak which is defined by the above Requirement (II) were calculated.

[Oxidation stability test]

[0203] The oxidation stability of the prepared grease composition was measured in accordance with JIS K2220 12:2013.

[0204] Specifically, 4.00 g of the prepared grease composition was distributed onto each of five sample dishes, a sample including the five sample dishes was placed in a system, and oxygen was enclosed therein at 685 kPa. Then, after adjusting the temperature to 99°C and the oxygen pressure to 755 kPa, the pressure drop was recorded every 24 hours, and the decrease in oxygen pressure after 100 hours was read and taken as the value of the oxidation stability.

[Fretting wear test]

[0205] Using the prepared grease composition in accordance with ASTM D4170, oscillation operation was performed under the following conditions, and the amount of wear (the amount of mass loss due to fretting wear) was measured.

- Bearings: Thrust bearing 51203
- Load: 2940N
- Oscillation angle: ± 0.105 rad
- Oscillation cycle: 25 Hz
- Time: 22 h
- Temperature: Room temperature (25°C)
- Enclosure amount of grease composition: 1.0 g per bearing set

[SRV test]

[0206] In the case of using each of the prepared grease compositions, the friction coefficient was measured with a SRV tester (manufactured by Optimol Instruments) under the following conditions.

- Cylinder: SUJ-2 material
- Disc: SUJ-2 material
- Frequency: 50 Hz
- Amplitude: 1.0 mm
- Load: 200N
- Temperature: 25°C
- Test time: 30 minutes

Table 1

				Example 1	Comparative Example 1
Formulation of Grease composition	Base oil	PAO	mass%	87.30	87.30
	Thickener	Urea-based thickener	mass%	9.70	9.70
	Antioxidant	44-dinonyldiphenylamine	mass%	2.00	2.00
	Rust inhibitor	alkenyl succinic acid polyhydric alcohol ester	mass%	1.00	1.00
	Total	-	mass%	100	100
Particle size distribution of particles containing urea-based thickener	Requirement (I)	Particle size indicating the maximum frequency of the peak	μm	0.6	90
	Requirement (II)	Half width of the peak	μm	0.6	30
Evaluation of various physical properties	Worked penetration (25°C)		-	273	265
	Dropping point		°C	260 or more	260 or more
	Oxidation stability test		kPa	25	25
	Fretting wear test, amount of wear		mg	4.00	37.00
	SRV test, friction coefficient		-	0.07	0.15

[0207] The grease composition prepared in Example 1 was excellent in anti-wear property and friction characteristics compared to Comparative Example 1.

[0208] Fig. 4 is a particle size distribution curve obtained by measuring the particle size of particles containing a urea-based thickener (B) in the grease composition produced in Example 1 on a volume basis with light scattering. In the particle size distribution curve shown in Fig. 4, the particle size r_1 of the peak P_1 indicating the maximum frequency y_1 was 0.6 μm , the half width x_1 of the peak P_1 was 0.6 μm , and Requirements (I) and (II) were satisfied.

[0209] On the other hand, Fig. 5 is a particle size distribution curve obtained by measuring particle size of particles containing the urea-based thickener (B) in the grease composition produced in Comparative Example 1 on a volume basis with light scattering.

[0210] In the particle size distribution curve shown in Fig. 5, the particle size r_2 of the peak P_2 indicating the maximum frequency y_2 was 90 μm , the half width x_2 of the peak P_2 was 30 μm , and therefore, Requirements (I) and (II) are not satisfied.

[0211] That is, in the grease composition prepared in Example 1 satisfying Requirements (I) and (II), even when the grease composition is mixed with an antioxidant or a rust inhibitor, aggregation of the urea-based thickener is suppressed, and it can be said that the urea-based thickener is highly dispersed. Therefore, it is considered that the anti-wear property and the friction reduction effect are improved while maintaining good oxidation stability.

Reference Signs List

[0212]

- 1 Grease manufacturing apparatus
- 2 Container body
- 3 Rotor
- 4 Introduction portion
- 4A, 4B Solution introducing pipe
- 5 Retention portion
- 6 First inner peripheral surface of container body
- 7 Second inner peripheral surface of container body
- 8 Discharge portion

- 9 First concave-convex portion on container side
- 10 Second concave-convex portion on container side
- 11 Discharge port
- 12 Rotation axis
- 5 13 First concave-convex portion of rotor
- 13A Concave portion
- 13B Convex portion
- 14 Second concave-convex portion of rotor
- 15 Scraper
- 10 A1, A2 Gap

Claims

- 15 1. A grease composition comprising: a base oil (A), a urea-based thickener (B), an antioxidant (C), and a rust inhibitor (D), wherein in a particle size distribution curve obtained by measuring the particle size of particles containing the urea-based thickener (B) in the grease composition on a volume basis with light scattering, a peak indicating the maximum frequency satisfies the following Requirements (I) and (II):

- 20 (I) a particle size indicating the maximum frequency of the peak is 1.0 μm or less; and
(II) a half width of the peak is 1.0 μm or less.

- 25 2. The grease composition according to claim 1, wherein the content of component (B) is from 1 to 40% by mass based on the whole amount of the grease composition.

3. The grease composition according to claim 1 or 2, wherein the content of component (C) is from 0.01 to 15% by mass based on the whole amount of the grease composition.

- 30 4. The grease composition according to any one of claims 1 to 3, wherein the content of component (D) is from 0.01 to 5% by mass based on the whole amount of the grease composition.

5. The grease composition according to any one of claims 1 to 4, wherein the kinematic viscosity of the base oil (A) at 40°C is from 10 to 130 mm^2/s .

- 35 6. The grease composition according to any one of claims 1 to 5, wherein the urea-based thickener (B) is a compound represented by the following general formula (b1):



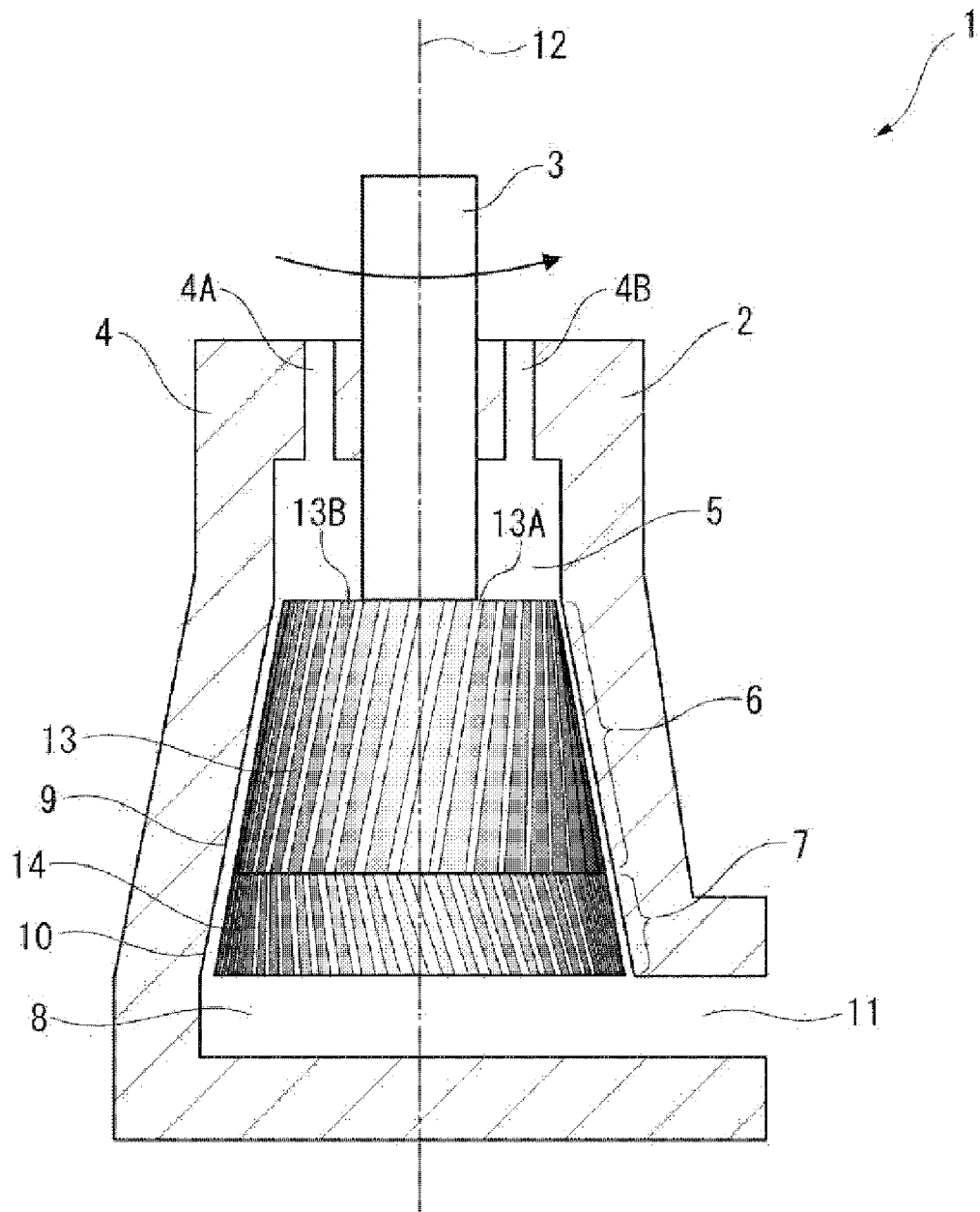
40 wherein R^1 and R^2 each independently represent a monovalent hydrocarbon group having 6 to 24 carbon atoms, and R^1 and R^2 may be the same as or different from each other, and R^3 represents a divalent aromatic hydrocarbon group having 6 to 18 carbon atoms.

- 45 7. The grease composition according to any one of claims 1 to 6, wherein the antioxidant (C) contains one or more kinds selected from an amine antioxidant (C1) and a phenol-based antioxidant (C2).

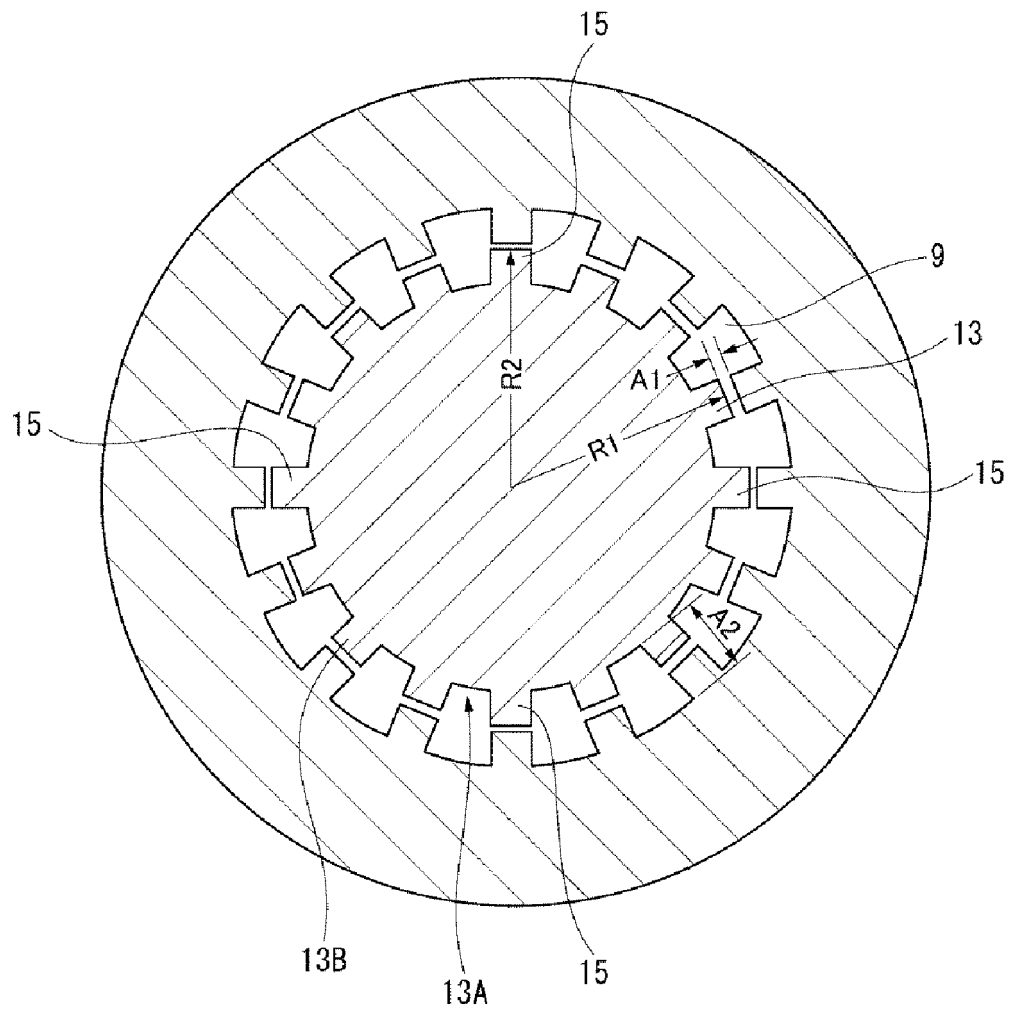
8. The grease composition according to any one of claims 1 to 7, wherein the rust inhibitor (D) contains an alkenyl succinic acid polyhydric alcohol ester.

- 50 9. The grease composition according to any one of claims 1 to 8, which is used in a bearing portion of a hub unit, an electric power steering, a driving electric motor flywheel, a ball joint, a wheel bearing, a spline portion, a constant velocity joint, a clutch booster, a servo motor, a blade bearing, or a generator.

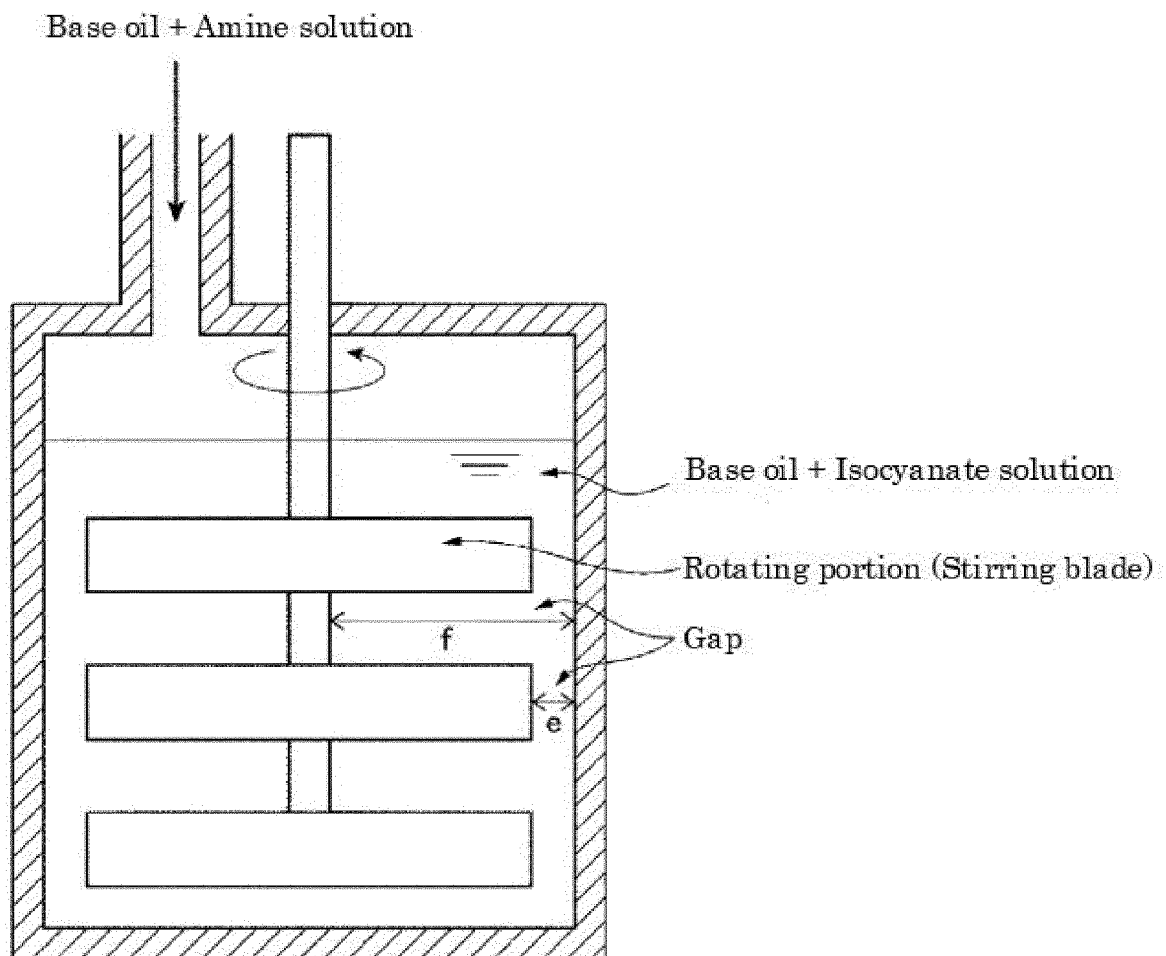
[Fig. 1]



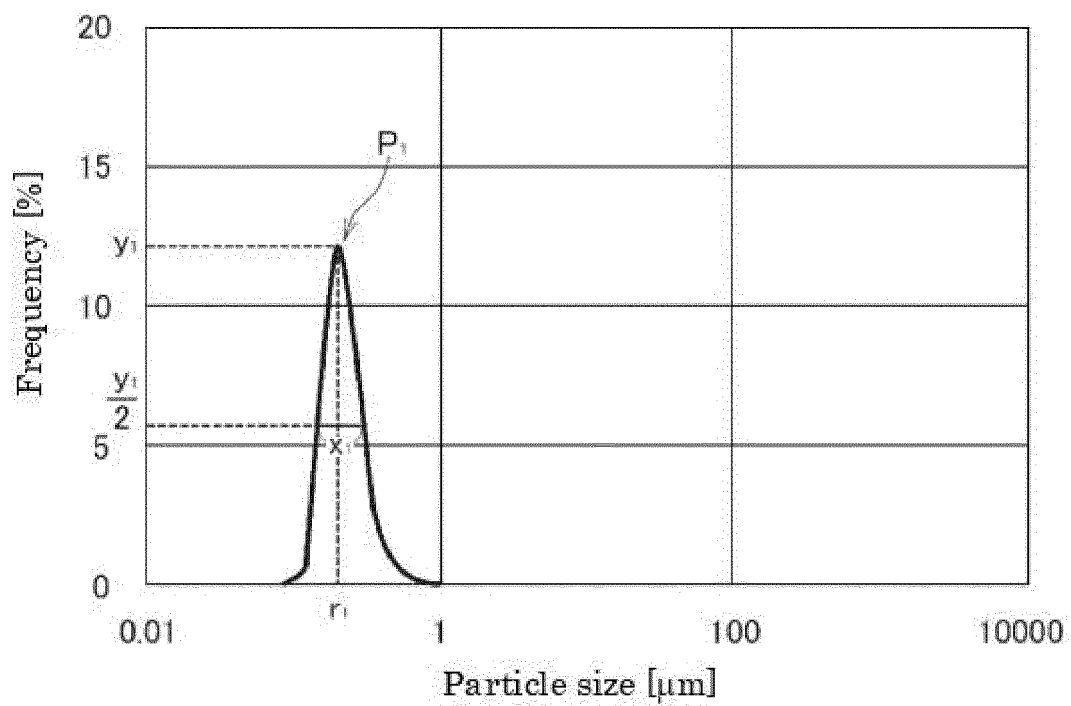
[Fig. 2]



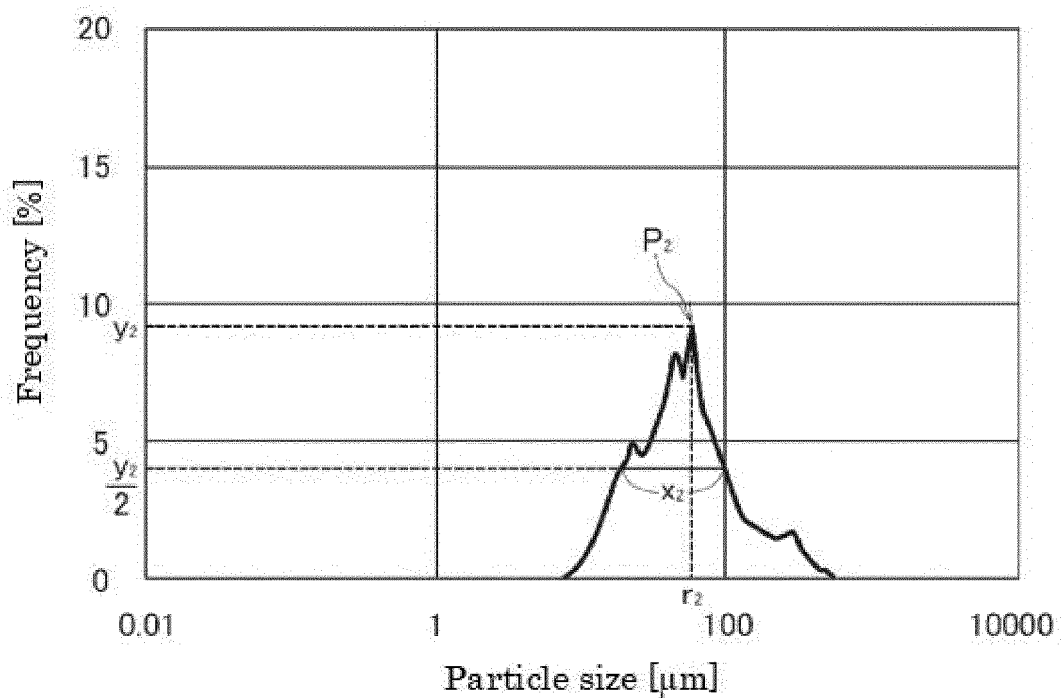
[Fig. 3]



[Fig. 4]



[Fig. 5]



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2018/030988

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl. C10M115/08 (2006.01) i, C10M129/10 (2006.01) i,
 C10M129/74 (2006.01) i, C10M133/04 (2006.01) i,
 C10N20/02 (2006.01) n, C10N20/06 (2006.01) n, C10N30/10 (2006.01) n,
 C10N30/12 (2006.01) n, C10N40/02 (2006.01) n, C10N40/04 (2006.01) n,
 C10N50/10 (2006.01) n

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl. C10M115/08, C10M129/10, C10M129/74, C10M133/04, C10N20/02,
 C10N20/06, C10N30/10, C10N30/12, C10N40/02, C10N40/04,
 C10N50/10

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
 Published unexamined utility model applications of Japan 1971-2018
 Registered utility model specifications of Japan 1996-2018
 Published registered utility model applications of Japan 1994-2018

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2017-115109 A (IDEMITSU KOSAN CO., LTD.) 29 June	1-9
Y	2017, claims, paragraphs [0025], [0029], [0031]- [0032], examples (Family: none)	1-9
Y	JP 2007-217609 A (NSK LTD.) 30 August 2007, paragraph [0043] (Family: none)	1-9



Further documents are listed in the continuation of Box C.



See patent family annex.

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2018/030988

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 2014/142198 A1 (IDEMITSU KOSAN CO., LTD.) 18 September 2014, examples & US 2016/0002558 A1, examples & EP 2975105 A1 & CN 105008503 A & KR 10-2015-0127673 A & TW 201443225 A	1-9
Y	JP 02-196894 A (SUMITOMO WIRING SYSTEMS, LTD.) 03 August 1990, examples & US 5370808 A, examples & EP 380094 A1	1-9
Y	JP 61-162595 A (NIPPON OIL CO., LTD.) 23 July 1986, examples (Family: none)	1-9
Y	JP 52-156274 A (NIPPON OIL CO., LTD.) 26 December 1977, examples & US 4115284 A, examples	1-9

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REFERENCES CITED IN THE DESCRIPTION

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