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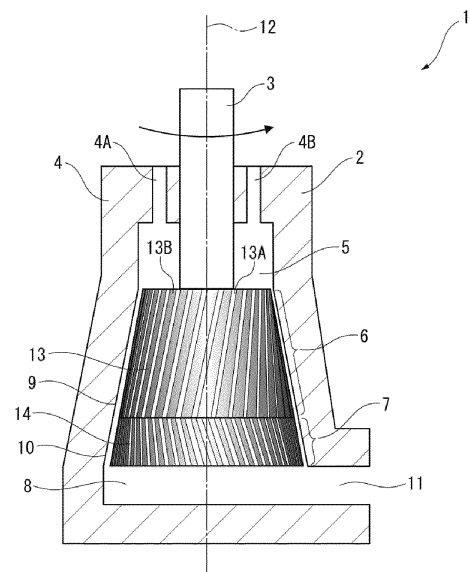
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(54) **LUBRICANT COMPOSITION AND METHOD FOR PRODUCING SAME**

(57) Provided a lubricant composition containing a lubricant base oil (A) and particles (B-1) containing a thickener (B), in which the particles (B-1) satisfy requirement (α): an arithmetic average particle diameter on a volume basis when the particles (B-1) are measured in an environment of 25°C by a laser diffraction/scattering method is 10.0 μm or less, and the lubricant composition satisfies requirement (β): a ratio $[(x1)/(x2)]$ of a shear viscosity (x1) at 40°C at a shear rate of 5 sec^{-1} and a shear viscosity (x2) at 40°C at a shear rate of 10,000 sec^{-1} is 2.0 or more and 100 or less.

Fig. 1



Description

Technical Field

5 **[0001]** The present invention relates to a lubricant composition and a method for producing the same.

Background Art

10 **[0002]** Oil retaining bearings formed by sintering metal powder are widely used as bearings incorporated in equipment such as automobile electrical equipment, home appliances, and OA office equipment.

[0003] The oil retaining bearing is generally produced by forming raw metal powder into a porous metal body through various steps such as mixing, molding, sintering, and sizing, and then vacuum impregnating the metal body with a lubricating oil composition using an impregnation apparatus, and is a sliding bearing used in a self-lubricated state.

15 **[0004]** The oil retaining bearing provides lubrication by supplying the lubricating oil composition impregnated into the porous metal body to a sliding surface between a rotating shaft and an inner surface of the bearing by a pumping action caused by rotation of the rotating shaft, and has not only excellent durability and rigidity, but also an advantage of keeping production costs low.

[0005] Various types of lubricating oil compositions used to be impregnated into the oil retaining bearing have been proposed in the past (see, for example, PTLs 1 and 2).

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Citation List

Patent Literature

25 **[0006]**

PTL 1: JP2005-179571A

PTL 2: JP2010-275471A

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Summary of Invention

Technical Problem

35 **[0007]** Incidentally, the lubricating oil composition used by being impregnated into the oil retaining bearing may be used with high viscosity by increasing viscosity of lubricant base oil or by adding a polymer viscosity improver, from the viewpoint of suppressing oil leakage and oil scattering. However, increasing viscosity of the lubricating oil composition causes an increase in stirring resistance during operation (during rotation).

40 **[0008]** Therefore, in the lubricating oil composition, there is a trade-off relationship between suppressing oil leakage and oil scattering and reducing stirring resistance during operation, and there is a problem that it is difficult to achieve both.

[0009] Note that this problem exists not only in the lubricating oil composition used by being impregnated into the oil retaining bearing, but also in lubricating oil for machine tools, and the like, which are required to suppress oil leakage and oil scattering.

45 **[0010]** Further, since the lubricating oil composition used by being impregnated into the oil retaining bearing is retained in pores existing in the oil retaining bearing, it is also required to have good pore permeability.

[0011] Note that pore permeability is required not only for the lubricating oil composition used by being impregnated into the oil retaining bearing, but also for the lubricating oil composition used in various devices and the like, in which oil is circulated and supplied, for example, through an oil filter. For example, in machine tools, the lubricating oil composition may be used by being circulated and supplied through the oil filter or the like.

50 **[0012]** In view of the above-mentioned problems, the present inventor has conducted extensive studies and has created a lubricant composition based on a new concept.

[0013] Therefore, an object of the present invention is to provide a lubricant composition capable of achieving both suppression of oil leakage and oil scattering and reduction of stirring resistance during operation, and excellent in pore permeability, and a method for producing the lubricant composition.

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Solution to Problem

[0014] According to the present invention, the following [1] to [3] are provided.

[1] A lubricant composition containing a lubricant base oil (A) and particles (B-1) containing a thickener (B), in which the particles (B-1) satisfy the following requirement (α), and the lubricant composition satisfies the following requirement (β).

- Requirement (α): An arithmetic average particle diameter on a volume basis when the particles (B-1) are measured in an environment of 25°C by a laser diffraction/scattering method is 10.0 μm or less.
- Requirement (β): A ratio $[(x1)/(x2)]$ of a shear viscosity ($x1$) at 40°C at a shear rate of 5 sec^{-1} and a shear viscosity ($x2$) at 40°C at a shear rate of 10,000 sec^{-1} is 2.0 or more and 100 or less.

[2] A method of using the lubricant composition according to the above [1] as impregnation bearing oil or lubricating oil for machine tools.

[3] A method for producing a lubricant composition, the method including a step of mixing:

a lubricant base oil (A); and
a grease (C) containing particles (B-2) containing a thickener (B), in which the particles (B-2) in the grease (C) satisfy the following requirement (γ).

- Requirement (γ): An arithmetic average particle diameter on a volume basis when the particles (B-2) are measured in an environment of 25°C by a laser diffraction/scattering method is 5.0 μm or less.

Advantageous Effects of Invention

[0015] According to the present invention, it is possible to provide a lubricant composition capable of achieving both suppression of oil leakage and oil scattering and reduction of stirring resistance during operation, and excellent in pore permeability, and a method for producing the lubricant composition.

Brief Description of Drawings

[0016]

Fig. 1 is a schematic diagram of a cross-section of a grease producing apparatus for miniaturizing particles (B-2) containing a thickener (B).

Fig. 2 is a schematic diagram of a cross-section of a first uneven portion of a container body of the grease producing apparatus of Fig. 1 in a direction perpendicular to a rotation axis.

Fig. 3 is a schematic diagram of a cross-section of a grease producing apparatus according to an embodiment different from that illustrated in Fig. 1, which is used in Comparative Production Examples 1 and 2.

Description of Embodiments

[0017] Upper and lower limits of numerical ranges described herein can be arbitrarily combined. For example, when "A to B" and "C to D" are described as the numerical ranges, the numerical ranges of "A to D" and "C to B" are also included in the scope of the present invention.

[0018] Further, the numerical range "lower limit to upper limit" described herein means that it is greater than or equal to the lower limit and less than or equal to the upper limit, unless otherwise specified.

[0019] Furthermore, in this description, numerical values in Examples are numerical values that can be used as the upper limit or the lower limit.

[Aspects of lubricant composition]

[0020] A lubricant composition of the present embodiment contains a lubricant base oil (A) and particles (B-1) containing a thickener (B).

[0021] Then, the particles (B-1) satisfy the following requirement (α), and the lubricant composition satisfies the following requirement (β).

- Requirement (α): An arithmetic average particle diameter on a volume basis when the particles (B-1) are measured in an environment of 25°C by a laser diffraction/scattering method is 10.0 μm or less.
- Requirement (β): A ratio $[(x1)/(x2)]$ of a shear viscosity ($x1$) at 40°C at a shear rate of 5 sec^{-1} and a shear viscosity ($x2$)

at 40°C at a shear rate of 10,000 sec⁻¹ is 2.0 or more and 100 or less.

[0022] The present inventor conducted extensive studies to solve the above problems. As a result, in order to achieve both suppression of oil leakage and oil scattering and reduction of stirring resistance during operation, the present inventor came up with an idea that it would be effective to create a lubricant composition that behaves like a so-called non-Newtonian fluid having low fluidity under low shear stress and high fluidity under high shear stress.

[0023] Further, in order to create a lubricant composition that has excellent pore permeability, the present inventor came up with an idea that there must be no particles in the lubricant composition, or even if there are particles, they must be extremely fine.

[0024] The present inventor conducted extensive studies based on these ideas. As a result, the present inventor has found that the lubricant composition containing the lubricant base oil (A) and the particles (B-1) containing the thickener (B) and satisfying the requirements (α) and (β) can solve the above problems, and has completed the present invention.

[0025] Here, first, the requirements (α) and (β) in the lubricant composition of the present embodiment will be described in detail.

<Requirement (α)>

[0026] The requirement (α) specifies that the arithmetic average particle diameter on the volume basis when the particles (B-1) containing the thickener (B) are measured in the environment of 25°C by the laser diffraction/scattering method is 10.0 μm or less.

[0027] In the requirement (α), "the particles (B-1) containing the thickener (B)" dispersed in the lubricant composition are to be measured. Measurement is performed by a method described in Examples described below.

[0028] Further, "the particles (B-1) containing the thickener (B)" refers to particles dispersed in the lubricant composition and formed by agglomerating the thickener (B).

[0029] When the arithmetic average particle diameter of the particles (B-1) containing the thickener (B) is more than 10.0 μm , since the particle diameter of the particles (B-1) in the lubricant composition is too large, a lubricant composition with poor permeability is formed.

[0030] Here, from the viewpoint of forming a lubricant composition with better pore permeability, the arithmetic average particle diameter of the particles (B-1) is preferably 9.5 μm or less.

[0031] Note that the lower limit of the arithmetic average particle diameter of the particles (B-1) is not particularly limited, but considering, for example, the viewpoint of facilitating preparation of the lubricant composition of the present embodiment, it is preferably 0.1 μm or more.

<Requirement (β)>

[0032] The requirement (β) specifies that the ratio $[(x1)/(x2)]$ of the shear viscosity ($x1$) at 40°C at the shear rate of 5 sec⁻¹ and the shear viscosity ($x2$) at 40°C at the shear rate of 10,000 sec⁻¹ is 2.0 or more and 100 or less.

[0033] When $[(x1)/(x2)]$ is less than 2.0, non-Newtonian fluid behavior of the lubricant composition weakens, and it is difficult to suppress the oil leakage and oil scattering.

[0034] Further, when $[(x1)/(x2)]$ is more than 100, it is difficult to reduce the stirring resistance during operation.

[0035] Here, from the viewpoint of making it easier to suppress the oil leakage and oil scattering of the lubricant composition, and from the viewpoint of making it easier to reduce the stirring resistance during operation, $[(x1)/(x2)]$ is preferably 2.5 or more, and more preferably 3.0 or more. Further, it is preferably 80 or less, more preferably 70 or less, still more preferably 60 or less, even more preferably 50 or less, even more preferably 40 or less, and even more preferably 30 or less.

[0036] Note that in this description, the shear viscosity means a value measured by the method described in Examples described below.

[0037] Next, constituent components of the lubricant composition of the present embodiment will be described in detail, also from the viewpoint of preparing a lubricant composition satisfying the requirements (α) and (β).

<Lubricant base oil (A)>

[0038] The lubricant composition of the present embodiment contains the lubricant base oil (A).

[0039] As the lubricant base oil (A), one or more selected from mineral oils and synthetic oils used in the past as a base oil for the lubricant composition can be used without particular limitation.

[0040] Examples of the mineral oils include: atmospheric residual oils obtained by atmospheric distillation of crude oil such as paraffinic crude oil, intermediate base crude oil, or naphthenic crude oil; distillate oils obtained by vacuum distillation of the atmospheric residual oils; and the mineral oils obtained by subjecting the distillate oils to one or more

refining treatments such as solvent deasphalting, solvent extraction, hydrocracking, solvent dewaxing, catalytic dewaxing, and hydrotreating.

[0041] Examples of the synthetic oils include: polyolefin oils such as 1-octene oligomers, 1-decene oligomers, hydrogenated products thereof, and ethylene- α -olefin copolymers; isoparaffin oils; various ester oils such as polyol esters and dibasic acid esters; various ether oils such as polyphenyl ethers; polyalkylene glycol oils; alkylbenzene oils; alkylnaphthalene oils; and GTL base oils obtained by isomerizing wax (gas-to-liquid (GTL) wax) produced from natural gas by the Fischer-Tropsch process or the like.

[0042] One of the mineral oils may be used alone, or two or more thereof may be used in combination. One of the synthetic oils may also be used alone, or two or more thereof may be used in combination. Further, one or more mineral oils and one or more synthetic oils may be used in combination.

[0043] Here, the lubricant base oil (A) preferably contains one or more selected from the mineral oils, the polyolefin oils, and the ester oils, for example, from the viewpoint of improving effects of the present invention. Among them, for example, from the viewpoint of further improving the effects of the present invention, the lubricant base oil (A) preferably contains an ester oil.

[0044] When the lubricant base oil (A) contains the ester oil, the lubricant base oil (A) may contain only the ester oil, but may further contain one or more selected from the group consisting of the mineral oils and the polyolefin oils in addition to the ester oil.

[0045] When the lubricant base oil (A) contains the ester oil, the content of the ester oil is preferably 10 mass% or more, more preferably 20 mass% or more, still more preferably 30 mass% or more, even more preferably 40 mass% or more, and even more preferably 50 mass% or more based on a total amount of the lubricant base oil (A).

[0046] Note that among the ester oils, dibasic acid ester oils are preferred.

[0047] From the viewpoint of facilitating the preparation of the lubricant composition satisfying the requirement (β), the lubricant base oil (A) preferably has a kinematic viscosity at 40°C (hereinafter also referred to as a "40°C kinematic viscosity") of 4.14 mm²/s or more, more preferably 9.00 mm²/s or more, and still more preferably 13.5 mm²/s or more. Further, the 40°C kinematic viscosity is preferably 110 mm²/s or less, and more preferably 74.8 mm²/s or less.

[0048] In this description, the 40°C kinematic viscosity of the lubricant base oil (A) means a value measured in accordance with JIS K2283:2000.

[0049] Note that when the lubricant base oil (A) is a mixed base oil containing two or more types of base oils, the 40°C kinematic viscosity of the mixed base oil is preferably within the above range.

[0050] In the present embodiment, from the viewpoint of facilitating the preparation of the lubricant composition satisfying the requirement (β), the content of the lubricant base oil (A) in the lubricant composition is preferably 80.0 mass% or more, and more preferably 85.0 mass% or more based on a total amount of the lubricant composition. Further, it is preferably 99.9 mass% or less, more preferably 99.0 mass% or less, and still more preferably 98.0 mass% or less.

[0051] The upper and lower limits of the numerical ranges can be arbitrarily combined. Specifically, it is preferably 80.0 mass% to 99.9 mass%, more preferably 85.0% to 99.0 mass%, and still more preferably 85.0% to 98.0 mass%.

[0052] Note that when the lubricant composition of the present embodiment is prepared by mixing the lubricant base oil (A) and the grease (C) as described below, the lubricant base oil (A) may include a base oil derived from the grease (C). A preferred range of the content of the lubricant base oil (A) including the base oil derived from the grease (C) is also the same as above.

<Particles (B-1) containing thickener (B)>

[0053] The lubricant composition of the present embodiment contains the particles (B-1) containing the thickener (B).

[0054] When the lubricant composition of the present embodiment does not contain the particles (B-1) containing the thickener (B), it cannot satisfy not only the requirement (α) but also the requirement (β).

[0055] In the lubricant composition of the present embodiment, inclusion of the particles (B-1) containing the thickener (B) is an important factor in satisfying the requirement (β).

[0056] Examples of the thickener (B) include various thickeners used in preparing grease compositions, such as one or more selected from the group consisting of soap-based thickeners and non-soap-based thickeners.

[0057] Examples of the soap-based thickeners include: metal soaps such as lithium soap, calcium soap, sodium soap, barium soap, and aluminum soap; and metal complex soaps such as lithium complex soap, calcium complex soap, barium complex soap, and aluminum complex soap.

[0058] Examples of the non-soap-based thickeners include urea-based thickeners, bentonite-based thickeners, and silica-based thickeners.

[0059] Here, from the viewpoint of facilitating the preparation of the lubricant composition satisfying the requirements (α) and (β) and further improving the effects of the present invention, the thickener (B) preferably includes the urea-based thickener.

[0060] Further, from the same viewpoint, the content of the urea-based thickener in the thickener (B) is preferably 50

mass% to 100 mass%, more preferably 60 mass% to 100 mass%, still more preferably 70 mass % to 100 mass %, even more preferably 80 mass % to 100 mass%, even more preferably 90 mass % to 100 mass %, and even more preferably 95 mass % to 100 mass % based on a total amount of the thickener (B).

[0061] Hereinafter, the urea-based thickener will be described in detail.

(Urea-based thickener)

[0062] The urea-based thickener may be any compound having a urea bond, but a diurea compound having two urea bonds is preferred, and a diurea compound represented by the following general formula (b 1) is more preferred.



[0063] Note that one of the urea-based thickeners may be used alone, or two or more thereof may be used in combination.

[0064] In the above general formula (b 1), R^1 and R^2 each independently represent a monovalent hydrocarbon group having 6 to 24 carbon atoms. R^1 and R^2 may be the same or different from each other. R^3 represents a divalent aromatic hydrocarbon group having 6 to 18 carbon atoms.

[0065] The monovalent hydrocarbon group that can be selected as R^1 and R^2 in the general formula (b 1) has 6 to 24 carbon atoms, but preferably 6 to 20 carbon atoms, and more preferably 6 to 18 carbon atoms.

[0066] Further, examples of the monovalent hydrocarbon groups that can be selected as R^1 and R^2 include saturated or unsaturated monovalent chain hydrocarbon groups (aliphatic hydrocarbon groups), saturated or unsaturated monovalent alicyclic hydrocarbon groups, and monovalent aromatic hydrocarbon groups.

[0067] Here, in R^1 and R^2 in the general formula (b 1), when the content of chain hydrocarbon groups is X molar equivalent, the content of alicyclic hydrocarbon groups is Y molar equivalent, and the content of aromatic hydrocarbon groups is Z molar equivalent, it is preferable that the following requirements (a) and (b) are satisfied.

- Requirement (a): A value of $[(X+Y)/(X+Y+Z)] \times 100$ is 90 or more (preferably 95 or more, more preferably 98 or more, and still more preferably 100).
- Requirement (b): X/Y ratio is 0/100 (X = 0, Y = 100) to 100/0 (X = 100, Y = 0) (preferably 10/90 to 90/10, and more preferably 15/85 to 85/15).

[0068] Note that since the chain hydrocarbon group, the alicyclic hydrocarbon group, and the aromatic hydrocarbon group are groups selected as R^1 and R^2 in the general formula (b 1), a sum of values of X, Y, and Z is 2 molar equivalents per 1 mol of the compound represented by the general formula (b1). Further, values of the requirements (a) and (b) mean average values based on a total amount of a compound group represented by the general formula (b 1) contained in the grease composition.

[0069] By using the compound represented by the general formula (b 1) that satisfies the requirements (a) and (b), the effects of the present invention can be more easily improved.

[0070] Note that the values of X, Y, and Z can be calculated from molar equivalents of amines used as raw materials.

[0071] Examples of monovalent saturated chain hydrocarbon groups that can be selected as R^1 and R^2 include linear or branched alkyl groups having 6 to 24 carbon atoms, and specifically hexyl group, heptyl group, octyl group, nonyl group, decyl group, undecyl group, dodecyl group, tridecyl group, tetradecyl group, pentadecyl group, hexadecyl group, heptadecyl group, octadecyl group, nonadecyl group, icosyl group, and the like.

[0072] Examples of monovalent unsaturated chain hydrocarbon groups that can be selected as R^1 and R^2 include linear or branched alkenyl groups having 6 to 24 carbon atoms, and specifically hexenyl group, heptenyl group, octenyl group, nonenyl group, decenyl group, undecenyl group, dodecenyl group, tridecenyl group, tetradecenyl group, pentadecenyl group, hexadecenyl group, heptadecenyl group, octadecenyl group, nonadecenyl group, icosenyl group, oleyl group, geranyl group, farnesyl group, linoleyl group, and the like.

[0073] Note that the monovalent saturated chain hydrocarbon group and the monovalent unsaturated chain hydrocarbon group may be linear or branched.

[0074] Examples of monovalent saturated alicyclic hydrocarbon groups that can be selected as R^1 and R^2 include: cycloalkyl groups such as cyclohexyl group, cycloheptyl group, cyclooctyl group, and 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 methylcyclohexyl group, dimethylcyclohexyl group, ethylcyclohexyl group, diethylcyclohexyl group, propylcyclohexyl group, isopropylcyclohexyl group, 1-methyl-propylcyclohexyl group, butylcyclohexyl group, pentylcyclohexyl group, pentyl-methylcyclohexyl group, and hexylcyclohexyl group.

[0075] Examples of monovalent unsaturated alicyclic hydrocarbon groups that can be selected as R^1 and R^2 include: cycloalkenyl groups such as cyclohexenyl group, cycloheptenyl group, and 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 methylcyclohexenyl group, dimethylcyclohexenyl group, ethylcyclohexenyl group, diethylcyclohexenyl group, and propylcyclohexenyl group.

[0076] Examples of the monovalent aromatic hydrocarbon groups that can be selected as R¹ and R² include phenyl group, biphenyl group, terphenyl group, naphthyl group, diphenylmethyl group, diphenylethyl group, diphenylpropyl group, methylphenyl group, dimethylphenyl group, ethylphenyl group, and propylphenyl group.

[0077] The divalent aromatic hydrocarbon group that can be selected as R³ in the general formula (b1) has 6 to 18 carbon atoms, but preferably 6 to 15 carbon atoms, and more preferably 6 to 13 carbon atoms.

[0078] Examples of the divalent aromatic hydrocarbon group that can be selected as R³ include phenylene group, diphenylmethylenylene group, diphenylethylenylene group, diphenylpropylenylene group, methylphenylenylene group, dimethylphenylenylene group, and ethylphenylenylene group.

[0079] Among them, phenylene group, diphenylmethylenylene group, diphenylethylenylene group, and diphenylpropylenylene group are preferred, and diphenylmethylenylene group is more preferred.

[0080] In the lubricant composition of the present embodiment, from the viewpoint of facilitating the preparation of the lubricant composition satisfying the requirement (β), the content of the thickener (B) is preferably 0.1 mass% or more, more preferably 0.2 mass% or more, and still more preferably 0.3 mass% or more based on the total amount of the lubricant composition. Further, it is preferably less than 2.0 mass%, more preferably 1.0 mass% or less, and still more preferably 0.8 mass% or less. When the content of the thickener (B) is within these ranges, the requirement (β) specified in the lubricant composition of the present embodiment can be more easily satisfied, and the effects of the present invention can be more easily improved.

[0081] Note that the upper and lower limits of the numerical ranges can be arbitrarily combined. Specifically, they are preferably 0.1 mass% or more and less than 2.0 mass%, more preferably 0.2 mass% to 1.0 mass%, and still more preferably 0.3 mass% to 0.8 mass%.

[0082] A method for producing the lubricant composition for making the particles (B-1) containing the thickener (B) satisfy the requirement (α) specified in the lubricant composition of the present embodiment is not particularly limited, and production methods applying various methods for miniaturizing the particles (B-1) containing the thickener (B) can be adopted as appropriate.

[0083] Here, in the final form of the lubricant composition of the present embodiment, the particles (B-1) containing the thickener (B) that have been miniaturized and adjusted to a specific particle diameter only need to be dispersed in the lubricant base oil (A) to be prepared. As an example, from the viewpoint of further facilitating the preparation of the lubricant composition satisfying the requirements (α) and (β) and further improving the effects of the present invention, the lubricant composition of the present invention is preferably produced by a production method described below.

[Method for producing lubricant composition]

[0084] An example of the method for producing the lubricant composition of the present embodiment is a production method including a step of mixing a lubricant base oil (A) and a grease (C) containing particles (B-2) containing a thickener (B).

[0085] Then in the production method, the particles (B-2) preferably satisfy the following requirement (γ).

- Requirement (γ): The arithmetic average particle diameter on the volume basis when the particles (B-2) are measured in the environment of 25°C by the laser diffraction/scattering method is 5.0 μm or less.

[0086] In the requirement (γ), "the particles (B-2) containing the thickener (B)" dispersed in the grease are to be measured. The measurement is performed by the method described in Examples described below.

[0087] Further, "the particles (B-2) containing the thickener (B)" refers to particles dispersed in the grease (C) and formed by agglomerating the thickener (B).

[0088] When the lubricant base oil (A) and the grease (C) containing the particles (B-2) containing the thickener (B) are mixed, "the particles (B-2) containing the thickener (B)" dispersed in the grease are dispersed in the lubricant base oil (A), to be "the particles (B-1) containing the thickener (B)" dispersed in the lubricant composition.

[0089] It is presumed that "the particles (B-1) containing the thickener (B)" swell slightly more than "the particles (B-2) containing the thickener (B)" due to an influence of compatibility with the lubricant base oil (A) and the like, to have the particle diameter specified by the requirement (α).

[0090] Therefore, the arithmetic average particle diameter of "the particles (B-2) containing the thickener (B)" specified in the requirement (γ) is preferably as small as possible from the viewpoint of facilitating the preparation of the lubricant composition satisfying the requirement (α) and improving the pore permeability of the lubricant composition.

[0091] Specifically, it is preferably 4.0 μm or less, more preferably 3.0 μm or less, and still more preferably 2.0 μm or less. Further, it is usually 0.01 μm or more.

[0092] Here, from the viewpoint of facilitating the preparation of the lubricant composition satisfying the requirements (α) and (β) and further improving the effects of the present invention, the thickener (B) in the grease (C) preferably contains the urea-based thickener.

[0093] Further, from the same viewpoint, the content of the urea-based thickener is preferably 50 mass% to 100 mass%, more preferably 60 mass% to 100 mass%, still more preferably 70 mass% to 100 mass%, even more preferably 80 mass% to 100 mass%, even more preferably 90 mass% to 100 mass%, and even more preferably 95 mass% to 100 mass% based on the total amount of the thickener (B).

[0094] An amount of the grease (C) to be blended is adjusted as appropriate so that the content of thickener (B) in the lubricant composition can be adjusted to the above-mentioned preferred range by considering the content of the thickener (B) in the grease (C).

[0095] The content of the thickener (B) in the grease (C) is, from the viewpoint of facilitating dispersion of the grease (C) in the lubricant base oil (A) and facilitating the preparation of the lubricant composition of the present embodiment, preferably 3 mass% to 20 mass%, more preferably 3 mass% to 15 mass%, still more preferably 3 mass% to 12 mass%, and even more preferably 3 mass% to 10 mass% based on a total amount of the grease (C).

[0096] Further, from the same viewpoint, a worked penetration of the grease (C) at 25°C is preferably 310 or more.

[0097] Here, from the viewpoint of facilitating preparation of the grease (C) containing the particles (B-2) satisfying the requirement (γ), the thickener (B) preferably contains the urea-based thickener, as described above. Then, the grease (C) is preferably produced, for example, by a production method described below.

<Method for producing grease (C)>

[0098] The urea-based thickener can usually be obtained by reacting an isocyanate compound with a monoamine. The reaction is preferably carried out by a method of adding a solution β in which the monoamine is dissolved in the base oil to a heated solution α obtained by dissolving the isocyanate compound in the base oil.

[0099] For example, when synthesizing the compound represented by the general formula (b1), a desired urea-based thickener can be synthesized by the above method using, as the isocyanate compound, an isocyanate compound having a group corresponding to the divalent aromatic hydrocarbon group represented by R^3 in the general formula (b1), and using, as the monoamine, an amine having a group corresponding to the monovalent hydrocarbon group represented by R^1 and R^2 .

[0100] Note that from the viewpoint of miniaturizing the particles (B-2) in the grease (C) so as to satisfy the requirement (γ), the grease (C) is preferably produced using a grease producing apparatus as illustrated in <1> below.

<1> A grease producing apparatus including:

a container body having an introduction portion into which a grease raw material is introduced and a discharge portion for discharging the grease to an 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, in which
the rotor includes a first uneven portion

- (i) having recess and protrusion alternately provided along a surface of the rotor, the recess and protrusion being inclined with respect to the rotation axis, and
- (ii) having a feeding ability from the introduction portion toward the discharge portion.

[0101] The grease producing apparatus described in the above <1> will be described below, and "preferred" provisions described below are aspects from the viewpoint of miniaturizing the particles (B-2) in the grease (C) so as to satisfy the requirement (γ) unless otherwise specified.

[0102] Fig. 1 is a schematic diagram of a cross-section of the grease producing apparatus of the above <1>, which can be used in the present embodiment.

[0103] The grease producing apparatus 1 illustrated in Fig. 1 includes a container body 2 into which the grease raw material is introduced, and a rotor 3 having a rotation axis 12 on a central axis line of the inner periphery of the container body 2 and rotating about the rotation axis 12.

[0104] The rotor 3 rotates at high speed about the rotation axis 12, and applies a high shear force to the grease raw material inside the container body 2. Thus, the grease (C) that satisfies the requirement (γ) is produced.

[0105] As illustrated in Fig. 1, the container body 2 is preferably divided into 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 in order from upstream.

[0106] As illustrated in Fig. 1, the container body 2 preferably has a truncated conical inner peripheral surface having an inner diameter gradually increasing from the introduction portion 4 toward the discharge portion 8.

[0107] The introduction portion 4, which is one end of the container body 2, includes a plurality of solution introduction pipes 4A and 4B that introduce the grease raw material from the outside of the container body 2.

[0108] The retention portion 5 is a space that is disposed downstream of the introduction portion 4 and temporarily retains the grease raw material introduced from the introduction portion 4. When the grease raw material is retained in the retention portion 5 for a long time, since the grease adhering to the inner peripheral surface of the retention portion 5 will form a large lump, it is preferable to be conveyed to the first inner peripheral surface 6 on the downstream side in as short a time as possible. More preferably, it is preferably conveyed directly to the first inner peripheral surface 6 without passing through the retention portion 5.

[0109] The first inner peripheral surface 6 is disposed at a downstream portion adjacent to the retention portion 5, and the second inner peripheral surface 7 is disposed at a downstream portion adjacent to the first inner peripheral surface 6. As will be described in detail later, providing a first uneven portion 9 on the first inner peripheral surface 6 and providing a second uneven portion 10 on the second inner peripheral surface 7 are preferable in order for the first inner peripheral surface 6 and the second inner peripheral surface 7 to function as a high shear portion that applies a high shear force to the grease raw material or the grease.

[0110] The discharge portion 8, which is the other end of the container body 2, is a portion that discharges the grease stirred on the first inner peripheral surface 6 and the second inner peripheral surface 7, and includes a discharge port 11 for discharging the grease. The discharge port 11 is formed in a direction perpendicular or substantially perpendicular to the rotation axis 12. Thus, the grease is discharged from the discharge port 11 in the direction perpendicular to or substantially perpendicular to the rotation axis 12. However, the discharge port 11 does not necessarily need to be perpendicular to the rotation axis 12, and may be formed in a direction parallel or substantially parallel to the rotation axis 12.

[0111] The rotor 3 is rotatably provided around the central axis line of the truncated conical inner peripheral surface of the container body 2 as the rotation axis 12, and rotates counterclockwise when the container body 2 is viewed from an upstream portion to a downstream portion as illustrated in Fig. 1.

[0112] The rotor 3 has an outer peripheral surface that expands as the inner diameter of the truncated cone of the container body 2 increases, 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 spaced at a constant distance.

[0113] The outer peripheral surface of the rotor 3 is provided with a first uneven portion 13 of the rotor, in which the recess and the protrusion are alternately provided along the surface of the rotor 3.

[0114] The first uneven portion 13 of the rotor is inclined with respect to the rotation axis 12 of the rotor 3 in a direction from the introduction portion 4 to the discharge portion 8, and has a feeding ability in the direction from the introduction portion 4 to the discharge portion 8. That is, the first uneven portion 13 of the rotor is inclined in a direction that extrudes the solution downstream when the rotor 3 rotates in a direction illustrated in Fig. 1.

[0115] A level difference between a recess 13A and a protrusion 13B of the first uneven 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 a diameter of the recess 13A of the outer peripheral surface of the rotor 3 is 100.

[0116] The number of protrusions 13B of the first uneven portion 13 of the rotor in a circumferential direction is preferably 2 to 1000, more preferably 6 to 500, and still more preferably 12 to 200.

[0117] A ratio [width of protrusion/width of recess] of a width of the protrusion 13B of the first uneven portion 13 of the rotor to a width of the recess 13A in a cross-section perpendicular 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.

[0118] An angle of inclination of the first uneven 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.

[0119] It is preferable that the first inner peripheral surface 6 of the container body 2 is provided with the first uneven portion 9 in which a plurality of unevennesses are formed along the inner peripheral surface.

[0120] Further, it is preferable that the unevennesses of the first uneven portion 9 of the container body 2 are inclined in an opposite direction to those of the first uneven portion 13 of the rotor.

[0121] That is, it is preferable that the plurality of unevennesses of the first uneven portion 9 of the container body 2 are inclined in the direction that extrudes the solution downstream when the rotor 3 rotates about the rotation axis 12 in the direction illustrated in Fig. 1. The first uneven portion 9 having the plurality of unevennesses provided on the first inner peripheral surface 6 of the container body 2 further enhances a stirring ability and a discharging ability.

[0122] A depth of the unevenness of the first uneven portion 9 of the container body 2 is preferably 0.2 to 30, more preferably 0.5 to 15, and still more preferably 1 to 5 when the inner diameter of the container is 100.

[0123] The number of unevennesses in the first uneven portion 9 of the container body 2 is preferably 2 to 1000, more preferably 6 to 500, and still more preferably 12 to 200.

[0124] A ratio [width of recess/width of protrusion] of the width of the recess of the unevenness of the first uneven portion 9 of the container body 2 to the width of the protrusion between grooves is preferably 0.01 to 100, more preferably is 0.1 to 10, and still more preferably 0.5 to 2 or less.

[0125] An angle of inclination of the unevenness of the first uneven portion 9 of the container body 2 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.

[0126] Note that by providing the first uneven portion 9 on the first inner peripheral surface 6 of the container body 2, the first inner peripheral surface 6 can function as a shearing portion that applies a high shear force to the grease raw material or the grease, but the first uneven portion 9 may not necessarily be provided.

[0127] It is preferable that a second uneven portion 14 of the rotor in which the recess and the protrusion are alternately provided along the surface of the rotor 3 is provided on an outer peripheral surface of a downstream portion of the first uneven portion 13 of the rotor.

[0128] The second uneven portion 14 of the rotor is inclined with respect to the rotation axis 12 of the rotor 3, and has an ability to suppress feeding from the introduction portion 4 to the discharge portion 8 to push the solution back upstream.

[0129] The level difference of the second uneven 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 recess of the outer peripheral surface of the rotor 3 is 100.

[0130] The number of protrusions of the second uneven 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.

[0131] The ratio [width of protrusion/width of recess] of the width of the protrusion to the width of the recess of the second uneven portion 14 of the rotor in the cross-section perpendicular to the rotation axis of the rotor 3 is preferably 0.01 to 100, more preferably 0.1 to 10, and still more preferably 0.5 to 2.

[0132] An angle of inclination of the second uneven 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.

[0133] It is preferable that the second inner peripheral surface 7 of the container body 2 is provided with the second uneven portion 10 having a plurality of unevennesses formed adjacent downstream of the unevenness in the first uneven portion 9 of the container body 2.

[0134] It is preferable that the plurality of the unevennesses are formed on the inner peripheral surface of the container body 2, and each unevenness is inclined in a direction opposite to a direction of inclination of the second unevenness portion 14 of the rotor.

[0135] That is, it is preferable that the plurality of unevennesses of the second uneven portion 10 of the container body 2 are inclined in a direction of pushing the solution back upstream when the rotor 3 rotates about the rotation axis 12 in the direction illustrated in Fig. 1. The stirring ability is further enhanced by the unevenness of the second uneven 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 shearing portion that applies a high shear force to the grease raw material or the grease.

[0136] A depth of the recess of the second uneven portion 10 of the container body 2 is preferably 0.2 to 30, more preferably 0.5 to 15, and still more preferably 1 to 5 when the inner diameter of the container body 2 is 100.

[0137] The number of recesses of the second uneven portion 10 of the container body 2 is preferably 2 to 1000, more preferably 6 to 500, and still more preferably 12 to 200.

[0138] The ratio [width of protrusion/width of recess] of the width of the protrusion and the width of the recess of the unevenness of the second uneven portion 10 of the container body 2 in the cross-section perpendicular 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.

[0139] An angle of inclination of the second uneven portion 10 of the container body 2 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.

[0140] A ratio [length of first uneven portion/length of second uneven portion] of a length of the first uneven portion 9 of the container body 2 and a length of the second uneven portion 10 of the container body 2 is preferably 2/1 to 20/1.

[0141] Fig. 2 is a diagram of a cross-section of the first uneven portion 9 of the container body 2 of the grease producing apparatus 1 in the direction perpendicular to the rotation axis 12.

[0142] The first uneven portion 13 of the rotor illustrated in Fig. 2 is provided with a plurality of scrapers 15 having tips protruding closer to the inner peripheral surface of the container body 2 than tips in a protruding direction of the protrusion 13B of the first uneven portion 13. Further, although not illustrated, the second uneven portion 14 is also provided with a plurality of scrapers having tips of the protrusions protruding toward the inner peripheral surface of the container body 2, similarly to the first uneven portion 13.

[0143] The scraper 15 is for scraping off the grease adhering to the inner peripheral surface of the first uneven portion 9 of the container body 2 and the second uneven portion 10 of the container body 2.

[0144] As for an amount of protrusion of the tip of the scraper 15 relative to the amount of protrusion of the protrusion 13B of the first uneven portion 13 of the rotor, a ratio $[R2/R1]$ of a radius (R2) of the tip of the scraper 15 and a radius (R1) of the tip of the protrusion 13B is preferably more than 1.005 and less than 2.0.

[0145] The number of scrapers 15 is preferably 2 to 500, more preferably 2 to 50, and still more preferably 2 to 10.

[0146] Note that although the grease producing apparatus 1 illustrated in Fig. 2 is provided with the scraper 15, the scraper 15 may not be provided, or the scraper 15 may be provided intermittently.

[0147] To produce the grease (C) using the grease producing apparatus 1, the solution α and the solution β , which are grease raw materials described above, are respectively introduced from solution introduction pipes 4A and 4B of the

introduction portion 4 of the container body 2, and the rotor 3 is rotated at high speed, so that the grease (C), in which the thickener (B) is the urea-based thickener and the particles (B-2) are miniaturized to satisfy the requirement (γ), can be produced.

[0148] 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 usually 10^7 s^{-1} or less.

[0149] Further, a ratio (Max/Min) of a maximum shear rate (Max) to a minimum shear rate (Min) in shearing when the rotor 3 rotates at high speed is preferably 100 or less, more preferably 50 or less, and still more preferably 10 or less.

[0150] When the shear rate is as uniform as possible for a mixed solution, the urea-based thickener and its precursor in the grease (C) can be easily miniaturized, resulting in a more uniform grease structure.

[0151] Here, the maximum shear rate (Max) is a highest shear rate applied to the mixed solution, the minimum shear rate (Min) is a lowest shear rate applied to the mixed solution, and they are defined as below.

- Maximum shear speed (Max) = (linear speed at tip of protrusion 13B of first uneven portion 13 of rotor)/(gap A1 between tip of protrusion 13B of first uneven portion 13 of rotor and protrusion of first uneven portion 9 of first inner peripheral surface 6 of container body 2)
- Minimum shear speed (Min) = (linear speed of recess 13A of first uneven portion 13 of rotor)/(gap A2 between recess 13A of first uneven portion 13 of rotor and recess of first uneven portion 9 of first inner peripheral surface 6 of container body 2)

[0152] Note that the gap A1 and the gap A2 are as illustrated in Fig. 2.

[0153] Since the grease producing apparatus 1 includes the scraper 15, it is possible to scrape off the grease adhering to the inner peripheral surface of the container body 2, thereby preventing formation of lumps during kneading, and continuously producing the grease (C) in a short time, in which the urea-based thickener is miniaturized to satisfy the requirement (γ).

[0154] In addition, since the scraper 15 can prevent the retained grease from being resistance to rotation of the rotor 3 by scraping off the adhered grease, rotational torque of the rotor 3 can be reduced, thereby reducing power consumption of a driving source to produce the grease efficiently and continuously.

[0155] Since the inner peripheral surface of the container body 2 has a truncated conical shape having an inner diameter increasing from the introduction portion 4 to the discharge portion 8, a centrifugal force has an effect of discharging the grease or the grease raw material in a downstream direction, and the rotational torque of the rotor 3 can be reduced to continuously produce the grease.

[0156] The first uneven portion 13 of the rotor is provided on the outer peripheral surface of the rotor 3, the first uneven portion 13 of the rotor is inclined with respect to the rotation axis 12 of the rotor 3 and has the feeding ability from the introduction portion 4 to the discharge portion 8, the second uneven portion 14 of the rotor is inclined with respect to the rotation axis 12 of the rotor 3 and has the ability to suppress feeding from the introduction portion 4 to the discharge portion 8, and thus a high shear force can be applied to the solution, and the urea-based thickener in the grease (C) can be miniaturized so as to satisfy the requirement (γ).

[0157] Since the first uneven portion 9 is formed on the first inner peripheral surface 6 of the container body 2 and is inclined in the opposite direction to the first uneven portion 13 of the rotor, in addition to the effect of the first uneven portion 13 of the rotor, the grease raw material can be sufficiently stirred while extruding the grease or the grease raw material in the downstream direction, and even after blending an additive, the urea-based thickener in the grease (C) can be miniaturized so as to satisfy the requirement (γ).

[0158] In addition, since the second uneven portion 10 is provided on the second inner peripheral surface 7 of the container body 2 and the second uneven portion 14 of the rotor is provided on the outer peripheral surface of the rotor 3, it is possible to prevent the grease raw material from flowing out from the first inner peripheral surface 6 of the container body more than necessary, and thus the grease raw material can be highly dispersed by applying a high shear force to the solution, and the urea-based thickener in the grease (C) can be miniaturized so as to satisfy the requirement (γ).

<Other additives>

[0159] The lubricant composition of the present embodiment may or may not include other components (hereinafter also referred to as "other additives") other than the lubricant base oil (A) and the thickener (B) to the extent that the effects of the present invention are not significantly impaired.

[0160] Examples of the other additives include oiliness agents, antioxidants, rust inhibitors, metal deactivators, corrosion inhibitors, extreme pressure agents, antifoaming agents, solid lubricants, dispersants, viscosity index improvers, and pour point depressants.

[0161] One of these may be used alone, or two or more thereof may be used in combination.

[0162] In the lubricant composition of the present embodiment, a total content of other additives is preferably 0 mass% to

20 mass%, more preferably 0.1 mass% to 15 mass%, still more preferably 0.5 mass% to 10 mass%, and even more preferably 0.5 mass% to 5 mass% based on the total amount of the lubricant composition.

[Physical properties of lubricant composition]

<Shear viscosity and the like>

[0163] In the lubricant composition of the present embodiment, it is preferable that the shear viscosity at various temperatures and various ratios related to the shear viscosity at various temperatures satisfy requirements specified below.

(Ratio of shear viscosity (25°C) at shear rate of 5 s⁻¹ to shear viscosity (25°C) at shear rate of 10,000 s⁻¹)

[0164] In the lubricant composition of the present embodiment, the ratio of the shear viscosity (25°C) at the shear rate of 5 s⁻¹ to the shear viscosity (25°C) at the shear rate of 10,000 s⁻¹ is preferably 1.5 or more, more preferably 2.0 or more, and still more preferably 2.3 or more. Further, it is preferably 90 or less, more preferably 70 or less, still more preferably 60 or less, even more preferably 50 or less, even more preferably 40 or less, even more preferably 30 or less, and even more preferably 20 or less.

(Ratio of shear viscosity (100°C) at shear rate of 5 s⁻¹ to shear viscosity (100°C) at shear rate of 10,000 s⁻¹)

[0165] The lubricant composition of the present embodiment has a ratio of shear viscosity (100°C) at a shear rate of 5 s⁻¹ to shear viscosity (100°C) at a shear rate of 10,000 s⁻¹ of preferably 3.5 or more, more preferably 4.0 or more, and still more preferably 4.5 or more. Further, it is preferably 110 or less, more preferably 90 or less, still more preferably 80 or less, even more preferably 70 or less, even more preferably 60 or less, even more preferably 50 or less, even more preferably 40 or less.

<Pore permeability>

[0166] The lubricating oil composition of this example preferably has pore permeability of 50 mass% or more as measured by the method described in Examples described below.

[Applications of lubricant composition]

[0167] The lubricant composition of the present embodiment can achieve both suppression of oil leakage and oil scattering and reduction of stirring resistance during operation, and also has excellent pore permeability.

[0168] Therefore, the lubricant composition of the present embodiment can be used as various compositions that are required to achieve both suppression of oil leakage and oil scattering and reduction of stirring resistance during operation, and are also required to have excellent pore permeability.

[0169] Examples of such lubricant compositions include impregnation bearing oil. They also include lubricating oil for machine tools such as sliding surface oils.

[0170] Therefore, according to the present embodiment, a method of using the lubricant composition as the impregnation bearing oil or the lubricating oil for machine tools is provided.

[0171] Further, according to the present embodiment, use of the lubricating oil composition as the impregnation bearing oil or the lubricating oil for machine tools is provided.

[0172] Further, according to the present embodiment, an oil-impregnated bearing impregnated with the lubricant composition is provided.

[0173] Furthermore, according to the present embodiment, a machine tool including the lubricant composition is provided.

[One aspect of the present invention provided]

[0174] In one aspect of the present invention, the following [1] to [9] are provided.

[1] A lubricant composition containing a lubricant base oil (A) and particles (B-1) containing a thickener (B), in which

the particles (B-1) satisfy the following requirement (α), and
the lubricant composition satisfies the following requirement (β).

- Requirement (α): An arithmetic average particle diameter on a volume basis when the particles (B-1) are measured in an environment of 25°C by a laser diffraction/scattering method is 10.0 μm or less.
- Requirement (β): A ratio $[(x1)/(x2)]$ of a shear viscosity (x1) at 40°C at a shear rate of 5 sec^{-1} and a shear viscosity (x2) at 40°C at a shear rate of 10,000 sec^{-1} is 2.0 or more and 100 or less.

[2] The lubricant composition according to the above [1], in which the thickener (B) includes a urea-based thickener.

[3] The lubricant composition according to the above [1] or [2], in which a content of the thickener (B) is 0.1 mass% or more and less than 2.0 mass% based on a total amount of the lubricant composition.

[4] The lubricant composition according to any one of the above [1] to [3], which is used as impregnation bearing oil or lubricating oil for machine tools.

[5] A method of using the lubricant composition according to any one of the above [1] to [4] as impregnation bearing oil or lubricating oil for machine tools.

[6] A method for producing a lubricant composition, the method including a step of mixing:

a lubricant base oil (A); and

a grease (C) containing particles (B-2) containing a thickener (B), in which the particles (B-2) in the grease (C) satisfy the following requirement (γ).

- Requirement (γ): An arithmetic average particle diameter on a volume basis when the particles (B-2) are measured in an environment of 25°C by a laser diffraction/scattering method is 5.0 μm or less.

[7] The method for producing the lubricant composition according to the above [6], in which the thickener (B) includes a urea-based thickener.

[8] The method for producing the lubricant composition according to the above [6] or [7], in which a content of the thickener (B) in the grease (C) is 3 mass% or more and 20 mass% or less based on a total amount of the grease (C).

[9] The method for producing the lubricant composition according to any one of the above [6] to [8], in which the grease (C) has a worked penetration of 310 or more at 25°C.

[Examples]

[0175] The present invention will be specifically described using the following examples. However, the present invention is not limited to the following examples.

[Production Example 1 and Comparative Production Examples 1 to 4]

[0176] Grease (C)-1 (hereinafter also abbreviated as "grease (C)") and grease (C')-1 to grease (C')-4 (hereinafter also abbreviated as grease (C')) were prepared, which were raw materials for preparing the lubricant compositions of Examples 1 to 3 and Comparative Examples 1 to 6 by the method described in Production Example 1 and Comparative Production Examples 1 to 4.

(Base oils for grease preparation)

[0177] The following base oils were prepared as base oils for grease preparation.

[0178] Note that the 40°C kinematic viscosity of the base oils for grease preparation was measured in accordance with JIS K2283:2000.

- "Base oil for grease preparation (1)": A mixed base oil of low viscosity mineral oil (40°C kinematic viscosity: 18 mm^2/s) and high viscosity mineral oil (bright stock, 40°C kinematic viscosity: 400 mm^2/s). Low viscosity mineral oil: high viscosity mineral oil = 25:68 (mass ratio)
- "Base oil for grease preparation (2)": PAO (poly- α -olefin, kinematic viscosity at 40°C: 17 mm^2/s)
- "Base oil for grease preparation (3)": Low viscosity mineral oil (40°C kinematic viscosity: 90 mm^2/s)
- "Base oil for grease preparation (4)": High viscosity mineral oil (bright stock, kinematic viscosity at 40°C: 400 mm^2/s)

<Production Example 1: Preparation of grease (C)-1 (urea grease 1)>

[0179] The solution α was prepared by adding 3.16 parts by mass of diphenylmethane-4,4'-diisocyanate (MDI) to 46.93 parts by mass of the base oil for grease preparation (1) heated to 70°C.

[0180] Further, the solution β was prepared by adding 2.00 parts by mass of cyclohexylamine and 1.36 parts by mass of

octadecyl amine (stearylamine) to 46.55 parts by mass of the base oil for grease preparation (1) heated to 70°C, prepared separately.

[0181] Then, using the grease producing apparatus 1 illustrated in Fig. 1, equal amounts of the solution α heated to 70°C and the solution β heated to 70°C were simultaneously introduced into the container body 2 respectively from the solution introduction pipe 4A and the solution introduction pipe 4B, and the solution α and the solution β were continuously introduced into the container body 2 while the rotor 3 was being rotated. Thereafter, this mixture was heated to 160°C using a stirring apparatus illustrated in Fig. 3, stirred for 1 hour, and then homogenized by roll milling to prepare the grease (C)-1.

[0182] Note that rotation speed of the rotor 3 of the grease producing apparatus 1 used was 8,000 rpm. In addition, the maximum shear rate (Max) at this time was 10,500 s⁻¹, and stirring was performed at the ratio [Max/Min] of the maximum shear rate (Max) to the minimum shear rate (Min) of 3.5.

[0183] The urea-based thickener contained in the obtained grease (C)-1 corresponds to a compound in which R¹ and R² in the general formula (b1) are a cyclohexyl group or an octadecyl group (a stearyl group), and R³ is a diphenylmethylene group.

[0184] Furthermore, a molar ratio (octadecyl amine (aliphatic amine): cyclohexylamine (alicyclic amine)) of octadecyl amine (aliphatic amine) and cyclohexylamine (alicyclic amine) used as the raw materials is 2:8.

<Comparative Production Example 1: Preparation of grease (C')-1 (urea grease 2)>

[0185] The solution α was prepared by adding 2.18 parts by mass of diphenylmethane-4,4'-diisocyanate (MDI) to 33 parts by mass of the base oil for grease preparation (2) heated to 70°C.

[0186] Further, the solution β was prepared by adding 0.17 parts by mass of cyclohexylamine and 4.15 parts by mass of octadecyl amine (stearylamine) to 60.5 parts by mass of the base oil for grease preparation (2) heated to 70°C, prepared separately.

[0187] Then, using the grease producing apparatus illustrated in Fig. 3, the solution α heated to 70°C was introduced into the container body from the solution introduction pipe. Thereafter, while stirring, the solution β heated to 70°C was introduced from the solution introduction pipe into the container body containing the solution α . After all the solution β was introduced into the container body, the mixture was heated to 160°C while stirring was continued by rotating a stirring blade, held for 1 hour, and homogenized by roll milling to prepare the grease (C')-1.

[0188] Note that the maximum shear rate (Max) at this time was about 100 s⁻¹, and the minimum shear rate was 1.23 s⁻¹. Further, the ratio (Max/Min) of the maximum shear rate (Max) to the minimum shear rate (Min) was about 81.

[0189] The urea-based thickener contained in the grease (C')-1 corresponds to a compound in which R¹ and R² in the general formula (b1) are a cyclohexyl group or an octadecyl group (a stearyl group), and R³ is a diphenylmethylene group.

[0190] Furthermore, the molar ratio (octadecyl amine (aliphatic amine): cyclohexylamine (alicyclic amine)) of octadecyl amine (aliphatic amine) and cyclohexylamine (alicyclic amine) used as the raw materials is 9:1.

<Comparative Production Example 2: Preparation of grease (C')-2 (urea grease 3)>

[0191] The solution α was prepared by adding 3.17 parts by mass of diphenylmethane-4,4'-diisocyanate (MDI) to 33 parts by mass of the base oil for grease preparation (2) heated to 70°C.

[0192] Further, the solution β was prepared by adding 1.99 parts by mass of cyclohexylamine and 1.34 parts by mass of octadecyl amine (stearylamine) to 60.5 parts by mass of the base oil for grease preparation (2) heated to 70°C, prepared separately.

[0193] Then, using the grease producing apparatus illustrated in Fig. 3, the solution α heated to 70°C was introduced into the container body from the solution introduction tube. Thereafter, while stirring, the solution β heated to 70°C was introduced from the solution introduction pipe into the container body containing the solution α . After all the solution β was introduced into the container body, the mixture was heated to 160°C while stirring was continued by rotating a stirring blade, held for 1 hour, and homogenized by roll milling to prepare the grease (C')-2.

[0194] Note that the maximum shear rate (Max) at this time was about 100 s⁻¹, and the minimum shear rate was 1.23 s⁻¹. Further, the ratio (Max/Min) of the maximum shear rate (Max) to the minimum shear rate (Min) was about 81.

[0195] The urea-based thickener contained in the grease (C')-2 corresponds to a compound in which R¹ and R² in the general formula (b1) are a cyclohexyl group or an octadecyl group (a stearyl group), and R³ is a diphenylmethylene group.

[0196] Furthermore, the molar ratio (octadecyl amine (aliphatic amine): cyclohexylamine (alicyclic amine)) of octadecyl amine (aliphatic amine) and cyclohexylamine (alicyclic amine) used as the raw materials is 2:8.

<Comparative Production Example 3: Preparation of grease (C')-3 (lithium soap grease)>

[0197] A solution prepared by adding 6 parts by mass of 12-hydroxystearic acid to 28 parts by mass of the base oil for grease preparation (3) was heated while stirring, and when it reached 80°C, 4-fold aqueous solution of 0.9 parts by mass of

lithium hydroxide was added. While continuing stirring, this mixture was kept at 100°C and dehydrated. This mixture was further heated and held at 202°C for 10 minutes, and 55.58 parts by mass of the base oil for grease preparation (3) and 8.25 parts by mass of the base oil for grease preparation (4) were stirred and mixed, and homogenized by roll milling to prepare the grease (C')-3.

<Comparative Production Example 4: Preparation of grease (C')-4 (silica grease)>

[0198] 10 parts by mass of hydrophobic silica and 90 parts by mass of the base oil for grease preparation (2) were stirred and mixed, and homogenized by roll milling to prepare Grease (C')-4.

<Evaluation of grease (C) and grease (C')>

[0199] The following evaluations 1 and 2 were conducted for the grease (C) and the grease (C').

(Evaluation 1: Worked penetration and consistency number)

[0200] The worked penetration of the grease (C) and the grease (C') was measured at 25°C in accordance with JIS K2220:2013 (Clause 7).

[0201] In addition, consistency numbers of the grease (C) and the grease (C') were determined based on worked penetration values.

(Evaluation 2: Evaluation of average particle diameter of particles containing thickener)

[0202] For the grease (C) and the grease (C'), the arithmetic average particle diameter (corresponding to the requirement (γ)) of the particles (B-2) containing the thickener (B) was evaluated.

[0203] A measurement and analysis method was based on JIS Z8825. A high concentration sample measurement mode of a laser diffraction particle size analyzer (manufactured by Shimadzu Corporation, trade name: SALD-7500nano) was selected, and a measurement sample was directly measured without being diluted. First, for blank measurement, two glass slides were set in a sample chamber, and the blank measurement was performed. Next, a 2 mL dropper was filled with the measurement sample, and 0.1 mL of the sample was extruded from the dropper, placed on a surface of one glass slide used for the blank measurement, and sandwiched with the other glass slide. This was set in the laser diffraction particle size analyzer and the measurement was started to obtain a volume-based arithmetic average particle diameter.

[0204] Here, the "volume-based arithmetic average particle diameter" refers to an arithmetic average value of particle size distribution on a volume basis. The particle size distribution on a volume basis indicates frequency distribution of particle sizes in the entire particle to be measured, with a volume calculated from the particle size as a reference. Further, the arithmetic average value of the particle size distribution on a volume basis can be calculated using the following formulas (1) to (3).

[0205] First, a representative particle diameter in a particle size interval divided by logarithmic scale division is calculated using the formula (1), it is assumed that the total number of particles in a measurement interval is 100%, and an average value μ on a logarithmic scale is determined using the formula (2). Finally, the average particle diameter is determined using the following formula (3).

$$\text{Representative particle diameter in particle size interval} = \frac{\log_{10}x_j + \log_{10}x_{j+1}}{2} \quad (1)$$

$$\mu = \frac{1}{100} \sum_{j=1}^n q_j \left(\frac{\log_{10}x_j + \log_{10}x_{j+1}}{2} \right) \quad (2)$$

$$\text{Average particle diameter} = 10^{\mu} \quad (3)$$

[0206] In the above formula, j means a division number of the particle diameter. x_j means a maximum particle size in the j -th particle size range, and x_{j+1} means a minimum particle size. q_j is a relative particle amount (difference %) corresponding to the particle size interval $[x_j, x_{j+1}]$ ($J = 1, 2, 3, \dots, n$).

[0207] Table 1 shows details of formulations of the grease (C) and the grease (C') and evaluation results.

Table 1

	Type of grease	Base oil amount (mass%)	Thickener amount (mass%)	Aliphatic amine: alicyclic amin (molar ratio)	Worked penetration (25°C)	Consistency number (number)	Average particle diameter of particles containing thickener (μm) (corresponding to requirement (γ))
Production Example 1	Grease (C)-1 Urea grease 1	93.5	6.5	2:8	341	1	0.04
Comparative Production Example 1	Grease (C')-1 Urea grease 2	93.5	6.5	9:1	437	00	19.7
Comparative Production Example 2	Grease (C')-2 Urea grease 3	93.5	6.5	2:8	364	0	11.1
Comparative Production Example 3	Grease (C')-3 Lithium soap grease	94.4	5.6	-	413	00	21.0
Comparative Production Example 4	Grease (C')-4 Silica grease	90	10	-	419	00	12

[Examples 1 to 3 and Comparative Examples 1 to 6]

[0208] The lubricant base oil (A), and the grease (C) and the grease (C') prepared in Production Example 1 and Comparative Production Examples 1 to 4 were respectively mixed in formulations shown in Table 2, to prepare the lubricant compositions of Examples 1 to 3 and Comparative Examples 2 to 5.

[0209] In Comparative Example 1, only the lubricant base oil (A) was used.

[0210] In Comparative Example 6, polybutene (weight average molecular weight: 12,000) diluted with diluent oil (weight average molecular weight: 3,600) was mixed in the formulation shown in Table 2 instead of the grease.

(Lubricant base oil (A))

[0211] The lubricant base oil (A) used in Examples 1 to 3 and Comparative Examples 1 to 6 was as follows.

- "Lubricant base oil (A)-1": PAO (poly- α -olefin, 40°C kinematic viscosity: 30 mm²/s)
- "Lubricant base oil (A)-2": Ester oil (bis(2-ethylhexyl)dodecanedioic acid, 40°C kinematic viscosity: 14 mm²/s)

(Other additives)

[0212] Details of other additives blended in Example 3 are as follows.

- "Other additives": Phosphorous extreme pressure agents, phenolic antioxidants, amine antioxidants, dispersants, corrosion inhibitors

[0213] The following evaluations 3 to 5 were performed on the lubricant compositions of Examples 1 to 3 and Comparative Examples 1 to 6.

(Evaluation 3: Evaluation of average particle diameter of particles containing thickener)

[0214] The measurement sample was changed from the grease to the lubricant composition, and the average particle diameter (corresponding to the requirement (α)) of the particles containing the thickener was evaluated in the same manner as in Evaluation 2.

(Evaluation 4: Evaluation on shear viscosity)

[0215] The shear viscosity was measured using a stress-controlled rheometer (manufactured by Anton Paar, product name "MCR301"). The detailed measurement method is as follows.

[0216] About 1 mL of the lubricant composition was dropped onto a temperature-adjusted sample stage using the dropper, and the sample was sandwiched between 50 ϕ cone plates with a gap of 0.05 mm, and then measurement was started under the condition that a zero gap was set at a measurement temperature in advance. The lubricant composition of the present embodiment has thixotropic properties, and rheological properties such as shear viscosity may be affected depending on how the sample is sandwiched, and thus the sample was pre-sheared for 30 seconds under steady flow conditions and at a shear rate of 100 s⁻¹ in advance, and then allowed to stand for 3 minutes. Thereafter, the shear viscosity at 5 s⁻¹ and 10,000 s⁻¹ was obtained when the shear rate was increased logarithmically from 0.01 s⁻¹ to 10,000 s⁻¹ over 10 minutes.

[0217] Specifically, the shear viscosity was obtained under the following six conditions.

- Condition 1: Shear rate 5 s⁻¹, oil temperature 25°C
- Condition 2: Shear rate 10,000 s⁻¹, oil temperature 25°C
- Condition 3: Shear rate 5 s⁻¹, oil temperature 40°C
- Condition 4: Shear rate 10,000 s⁻¹, oil temperature 40°C
- Condition 5: Shear rate 5 s⁻¹, oil temperature 100°C
- Condition 6: Shear rate 10,000 s⁻¹, oil temperature 100°C

[0218] Then, based on the obtained measurement results, "(shear viscosity of condition 1)/(shear viscosity of condition 2)", "(shear viscosity of condition 3)/(shear viscosity of condition 4)", and "(shear viscosity of condition 5)/(shear viscosity of condition 6)" were calculated.

[0219] Note that "(shear viscosity of condition 3)/(shear viscosity of condition 4)" corresponds to the requirement (β).

(Evaluation 5: Evaluation of pore permeability by Millipore filter test)

[0220] Evaluation of the pore permeability was carried out by the following procedure.

[0221] An amount of filter residue M (mg/100 mL) (contaminant described in JIS B9931) is determined by a method based on JIS B9931: Method for measuring hydraulic oil contamination by mass method, and the pore permeability (mass%) was determined using the following formula (4). At this time, an amount of the sample was 5 mL, a solvent used for dilution was n-hexane, and the filter was a polycarbonate filter (manufactured by Merck KGaA, trade name: TCTP04700) with a pore size of 10 μ m.

$$\text{Pore permeability} = \left(1 - \frac{M}{1000\rho T_c} \right) \times 100 \quad (4)$$

[0222] In the formula (4), ρ is density (g/cm³) of the lubricant base oil (A), and T_c is the amount (parts by mass) of the thickener (B).

[0223] The evaluation criteria for pore permeability were as follows.

- Evaluation A: Pore permeability is 50 mass% or more
- Evaluation B: Pore permeability is less than 50 mass%

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[0224] In this example, the evaluation A was considered as passing.

[0225] The evaluation results are shown in Table 2.

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Table 2

Lubricant composition	Lubricant base oil (A)	Grease (C)	Grease (C')	Non-grease	Units	Example 1	Example 2	Example 3	Comparative Example 1	Comparative Example 2
Lubricant composition	(A)-1	PAO			mass%	92.31	-	-	10000	92.31
	(A)-2	Ester oil			mass%	-	92.31	89.66	-	-
	(C)-1	Urea grease 1			mass%	7.69	7.69	7.69	-	-
	(C')-1	Urea grease 2			mass%	-	-	-	-	7.69
	(C')-2	Urea grease 3			mass%	-	-	-	-	-
	(C')-3	Lithium soap grease			mass%	-	-	-	-	-
	(C')-4	Silica grease			mass%	-	-	-	-	-
		Polybutene			mass%	-	-	-	-	-
		Other additives			mass%	-	-	2.65	-	-
		Total			mass%	100.00	100.00	100.00	100.00	100.00
Evaluation results	Evaluation 3	Thickner in lubricating oil composition or resin content			mass%	0.5	0.5	0.5	-	0.5
		Average particle diameter of particles containing thickener (corresponding to requirement (α))			μm	5.5	8.4	3.9	-	19.9
	Evaluation 4	Condition 1	Shear viscosity (25°C, 5 s ⁻¹)		mPa·s	103.1	63.71	91.23	48.61	78.43
		Condition 2	Shear viscosity (25°C, 10000 s ⁻¹)		mPa·s	54.94	25.6	26.57	48.17	50.65
		Condition 3	Shear viscosity (40°C, 5 s ⁻¹)		mPa·s	63.71	48.90	69.82	29.95	44.46
		Condition 4	Shear viscosity (40°C, 10000 s ⁻¹)		mPa·s	29.97	15.97	16.60	26.45	27.64
		Condition 5	Shear viscosity (100°C, 5 s ⁻¹)		mPa·s	23.92	26.46	36.59	4.824	15.14
		Condition 6	Shear viscosity (100°C, 10000 s ⁻¹)		mPa·s	5.832	5.042	5.248	4.820	5.061

(continued)

	Condition 1/Condition 2	Shear viscosity ratio (25°C, 5 s ⁻¹ /10000 s ⁻¹)	Units	Example 1	Example 2	Example 3	Comparative Example 1	Comparative Example 2
		Condition 3/Condition 4 (corresponding to require- ment (β))	-	1.9	2.5	3.4	1.0	1.5
		Condition 5/Condition 6	-	2.1	3.1	4.2	1.1	1.6
			-	4.1	5.2	7.0	1.0	3.0
	Evaluation 5		-	A	A	A	A	B

Table 2 (continued)

Lubricant composition				Units	Comparative Example 3	Comparative Example 4	Comparative Example 5	Comparative Example 6	
Lubricant composition	Lubricant base oil (A)	(A)-1	PAO	mass%	92.31	91.00	95.00	99.26	
		(A)-2	Ester oil	mass%	-	-	-	-	
	Grease (C)	(C)-1	Urea grease 1	mass%	-	-	-	-	
		(C')-1	Urea grease 2	mass%	-	-	-	-	
		(C')-2	Urea grease 3	mass%	7.69	-	-	-	
	Grease (C')	(C')-3	Lithium soap grease	mass%	-	9.00	-	-	
		(C')-4	Silica grease	mass%	-	-	5.00	-	
		Non-grease	Polybutene	mass%	-	-	-	0.74	
	Evaluation results	Evaluation 3	Other additives		mass%	-	-	-	-
			Total		mass%	100.00	100.00	100.00	100.00
Thickner in lubricating oil composition or resin content			mass%	0.5	0.5	0.5	0.5		
Average particle diameter of particles containing thickener (corresponding to requirement (α))			μm	14.0	16.4	unmeasurable	-		
Evaluation results	Evaluation 4	Condition 1	Shear viscosity (25°C, 5 s ⁻¹)	mPa·s	99.33	127.4	59.32	52.75	
		Condition 2	Shear viscosity (25°C, 10000 s ⁻¹)	mPa·s	51.15	57.92	48.45	51.46	
		Condition 3	Shear viscosity (40°C, 5 s ⁻¹)	mPa·s	59.15	91.74	31.88	30.03	
		Condition 4	Shear viscosity (40°C, 10000 s ⁻¹)	mPa·s	28.39	31.34	26.48	28.84	
	Condition 5	Shear viscosity (100°C, 5 s ⁻¹)	mPa·s	32.07	63.81	6.255	7.582		
	Condition 6	Shear viscosity (100°C, 10000 s ⁻¹)	mPa·s	5.453	5.955	4.636	4.844		

(continued)

	Condition 1/Condition 2	Shear viscosity ratio (25°C, 5 s ⁻¹ /10000 s ⁻¹)	Units	Comparative Example 3	Comparative Example 4	Comparative Example 5	Comparative Example 6
		Condition 3/Condition 4 (corresponding to require- ment (β))	-	1.9	2.2	1.2	1.0
		Condition 5/Condition 6	-	2.1	2.9	1.2	1.0
			-	5.9	10.7	1.3	1.6
	Evaluation 5	Pore permeability		B	B	A	A

[0226] The following can be seen from Table 2.

[0227] The lubricant compositions of Examples 1 to 3 satisfy the requirement (α) and have excellent pore permeability.

[0228] Further, the lubricant compositions of Examples 1 to 3 also satisfy the requirement (β), and can achieve both suppression of oil leakage and oil scattering and reduction of stirring resistance during operation.

[0229] In contrast, the lubricant compositions of Comparative Examples 2 to 4 do not satisfy the requirement (α) and are therefore inferior in pore permeability.

[0230] Furthermore, the lubricant compositions of Comparative Examples 1, 2, 5, and 6 do not satisfy the requirement (β), and therefore cannot achieve both suppression of oil leakage and oil scattering and reduction of stirring resistance during operation.

Reference Signs List

[0231]

- 1: Grease producing apparatus
- 2: Container body
- 3: Rotor
- 4: Introduction portion
- 4A, 4B: Solution introduction pipe
- 5: Retention portion
- 6: First inner peripheral surface
- 7: Second inner peripheral surface
- 8: Discharge portion
- 9: First uneven portion of container body
- 10: Second uneven portion of container body
- 11: Discharge port
- 12: Rotation axis
- 13: First uneven portion of rotor
- 13A: Recess
- 13B: Protrusion
- 14: Second uneven portion of rotor
- 15: Scraper
- A1, A2: Gap.

Claims

1. A lubricant composition comprising a lubricant base oil (A) and particles (B-1) comprising a thickener (B), wherein
 - the particles (B-1) satisfy requirement (α): an arithmetic average particle diameter on a volume basis when the particles (B-1) are measured in an environment of 25°C by a laser diffraction/scattering method is 10.0 μm or less, and
 - the lubricant composition satisfies requirement (β): a ratio $[(x1)/(x2)]$ of a shear viscosity ($x1$) at 40°C at a shear rate of 5 sec^{-1} and a shear viscosity ($x2$) at 40°C at a shear rate of 10,000 sec^{-1} is 2.0 or more and 100 or less.
2. The lubricant composition according to claim 1, wherein the thickener (B) comprises a urea-based thickener.
3. The lubricant composition according to claim 1 or 2, wherein a content of the thickener (B) is 0.1 mass% or more and less than 2.0 mass% based on a total amount of the lubricant composition.
4. The lubricant composition according to any one of claims 1 to 3, which is used as impregnation bearing oil or lubricating oil for machine tools.
5. A method of using the lubricant composition according to any one of claims 1 to 4 as impregnation bearing oil or lubricating oil for machine tools.
6. A method for producing a lubricant composition, the method comprising a step of mixing:

a lubricant base oil (A); and
a grease (C) comprising particles (B-2) comprising a thickener (B), wherein
the particles (B-2) in the grease (C) satisfy requirement (γ): an arithmetic average particle diameter on a volume
basis when the particles (B-2) are measured in an environment of 25°C by a laser diffraction/scattering method is
5.0 μm or less.

7. The method for producing the lubricant composition according to claim 6, wherein the thickener (B) comprises a urea-based thickener.
8. The method for producing the lubricant composition according to claim 6 or 7, wherein a content of the thickener (B) in the grease (C) is 3 mass% or more and 20 mass% or less based on a total amount of the grease (C).
9. The method for producing the lubricant composition according to any one of claims 6 to 8, wherein the grease (C) has a worked penetration of 310 or more at 25°C.

Fig. 1

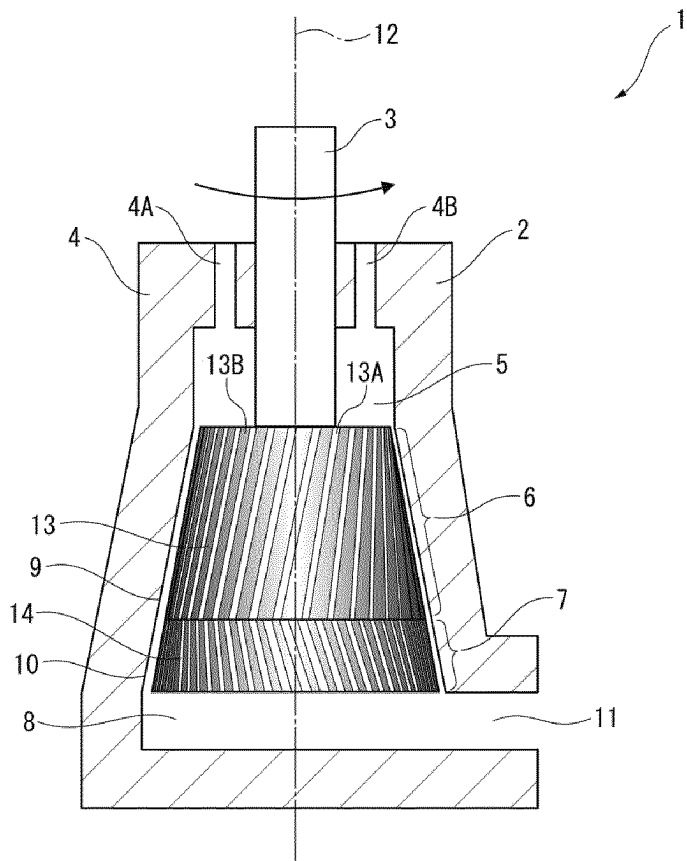


Fig. 2

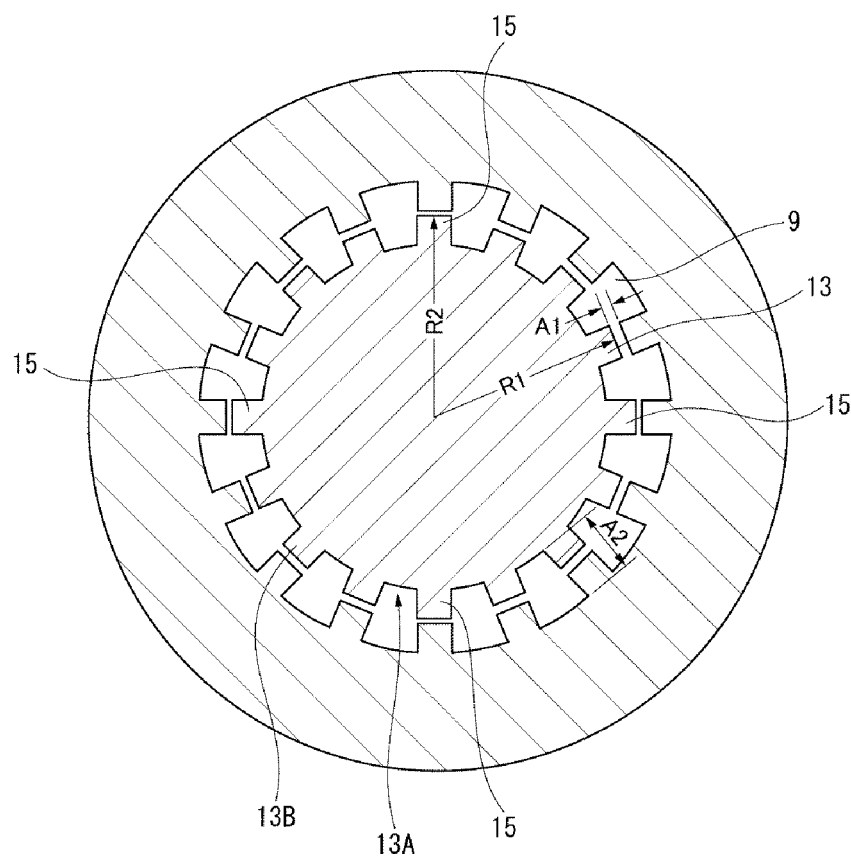
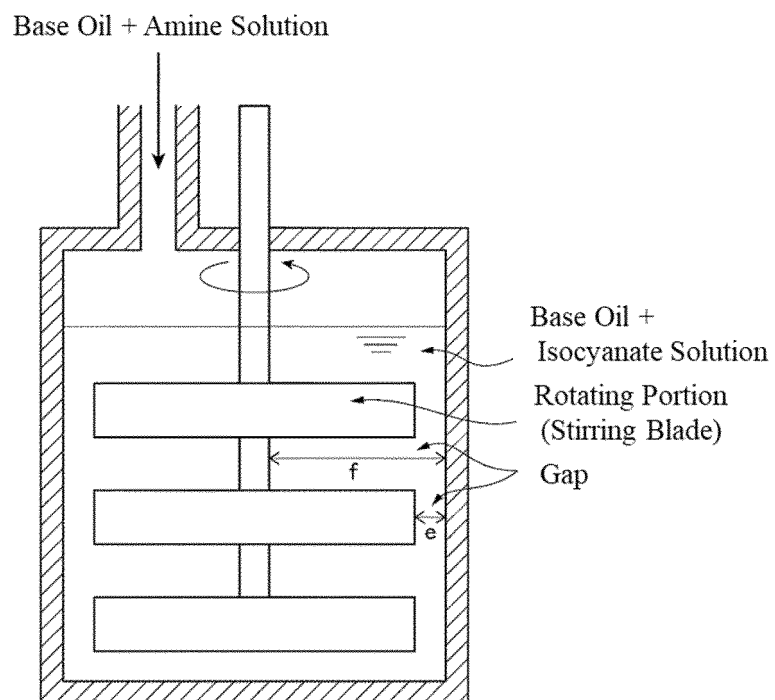


Fig. 3



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2023/012198

A. CLASSIFICATION OF SUBJECT MATTER

C10M 169/02(2006.01)i; **C10N 30/00**(2006.01)n; **C10N 40/02**(2006.01)n; **C10N 50/10**(2006.01)n; **C10M 115/08**(2006.01)i
FI: C10M169/02; C10M115/08; C10N50:10; C10N30:00 Z; C10N40:02

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)

C10M101/00-177/00; C10N30/00; C10N40/02; 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 1971-2023
Registered utility model specifications of Japan 1996-2023
Published registered utility model applications of Japan 1994-2023

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	WO 2021/200120 A1 (IDEMITSU KOSAN CO) 07 October 2021 (2021-10-07) paragraphs [0048], [0057], [0104]-[0113], table 1, examples 1-3	1-9
Y	paragraphs [0057], [0104]	3-5
X	JP 2016-145292 A (IDEMITSU KOSAN CO) 12 August 2016 (2016-08-12) paragraphs [0013], [0026], [0030], [0036], table 1, examples 3, 6	1-2, 6-9
Y	paragraphs [0013], [0026], [0030], [0036], table 1, examples 3, 6	3-5
A	JP 2012-13167 A (HITACHI POWDERED METALS CO LTD) 19 January 2012 (2012-01-19) entire text	1-9
A	JP 2016-204623 A (JTEKT CORP) 08 December 2016 (2016-12-08) entire text, all drawings	1-9

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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Information on patent family members

International application No.

PCT/JP2023/012198

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
WO 2021/200120 A1	07 October 2021	(Family: none)	
JP 2016-145292 A	12 August 2016	US 2018/0037842 A1 paragraphs [0036], [0134]- [0135], [0140], [0146], table 1, examples 3, 6	
JP 2012-13167 A	19 January 2012	(Family: none)	
JP 2016-204623 A	08 December 2016	(Family: none)	

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REFERENCES CITED IN THE DESCRIPTION

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- JP 2010275471 A [0006]