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(54) GREASE COMPOSITION

(57) A grease composition comprising
(a) a thickener containing a mixture of the following formulas (I), (II), and (III):

(I): R1-NHCONH-R2-NHCONH-R1,

(II): R3-NHCONH-R2-NHCONH-R3,

and

(III): R1-NHCONH-R2-NHCONH-R3,

where R1 is a cyclohexyl group, R2 is a C6-15 divalent aromatic hydrocarbon group, R3 is a C8-20 linear or branched alkyl group or alkenyl group, and {R1/(R1+R3)}×100 is 80 to 0 mol%, and
(b) a base oil,
wherein a 60-stroke worked penetration measured by a method specified in JIS K 2220.7 is 300 to 350, and
a penetration in a roll stability test measured after applying shearing at 120°C for 24 hours using a tester conforming to ASTM D 1831 is 440 or less.

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Description

Technical Field

[0001] The present invention relates to a grease composition that can be suitably used for lubricating portions that require shear stability of the grease, such as lubricating portions of machine parts having steel lubricated portions that perform rolling motion and rolling/sliding motion.

Background Art

[0002] Grease is a semi-solid or semi-liquid substance obtained by dispersing a thickener in a liquid lubricating oil (base oil). Grease, unlike lubricating oil, does not flow under an external force such as gravity; it begins to flow when a significant external force exceeding the yield value is applied, and when it begins to flow, it exhibits the same lubricating action as lubricating oil. This characteristic allows for miniaturization of equipment thanks to the advantage of simplifying the structure around the lubricating portion; therefore, grease is used in all industrial fields, including automobiles, electrical appliances, information equipment, railway vehicles, industrial machinery, and aerospace equipment.

[0003] The degree of semi-solid or solid state (hardness) that appears in the definition of grease is expressed by "consistency" and is graded according to the range of "worked penetration," which is the penetration after a specified number of reciprocating mixing cycles. When lubricating a machine part with grease, a grease with an appropriate consistency is selected from the viewpoints of ease of handling and leakage suppression. For example, for rolling bearing grease, No. 2 grade with a worked penetration of 265 to 295 is relatively often used; when supplying grease by centralized lubrication, No. 0 grade with a worked penetration of 355 to 385 is often used.

[0004] When grease is subjected to strong shearing at the lubrication portion, the three-dimensional network structure of the thickener in the grease is destroyed and cannot return to its original hardness, and generally softens. The degree of softening is called mechanical stability or shear stability and is one of the important practical characteristics.

Citation List

Non Patent Literatures

[0005]

Non Patent Literature 1: "Fundamentals and Applications of Lubricating Grease" edited by the Grease Research Committee of the Japan Society of Tribologists, P. 39 (2007), Yokendo
Non Patent Literature 2: "A Simple Story of Grease," P. 27 (2010), Juntsu Publishing Co., Ltd.

Summary of Invention

Problems to be solved by the invention

[0006] Excessive softening can cause problems such as leakage and scattering. In addition, this may lead to insufficient lubricant, causing damage to parts, such as seizure and peeling.

[0007] Furthermore, it is said that when the fluidity of the grease bulk inside the bearing is improved by softening, the exchange of grease between the retainer and the rolling element becomes uneven, and a phenomenon called churning occurs, in which a large amount of grease continues to exist in the running part where the rolling element travels, leading to increased churning torque, overheating, and further softening.

[0008] An object of the present invention is to provide a grease composition having excellent shear stability.

Means for solution of the problems

[0009]

1. A grease composition comprising

(a) a thickener containing a mixture of diurea compounds represented by the following formulas (I), (II), and (III):

(I): R1-NHCONH-R2-NHCONH-R1,

(II): R3-NHCONH-R2-NHCONH-R3,

and

(III): R1-NHCONH-R2-NHCONH-R3,

where R1 is a cyclohexyl group, R2 is a C6-15 divalent aromatic hydrocarbon group, R3 is a C8-20 linear or branched alkyl group or alkenyl group, and a ratio of the number of moles of the cyclohexyl group to the total number of moles of the cyclohexyl group and the alkyl group or alkenyl group [{R1/(R1+R3)}×100] is 80 to 0 mol%, and
(b) a base oil,

wherein a 60-stroke worked penetration measured by a method specified in JIS K 2220.7 is 300 to 350, and a penetration in a roll stability test measured after applying shearing at 120°C for 24 hours using a tester conforming to ASTM D 1831 is 440 or less.

2. The grease composition according to claim 1, wherein the diurea thickener exhibits a peak corresponding to the following formula (A) among peaks measured by X-ray diffraction:

[Molecular Cell Size of Diurea Thickener Measured by X-ray Diffraction] ÷ [Molecular Cell Size of the Diurea Thickener Obtained by Geometric Calculation] = 0.7 to 0.4 (A)

Advantageous Effects of Invention

[0010] The grease composition of the present invention has excellent shear stability.

Brief Description of Drawings

[0011] Fig. 1 illustrates an X-ray diffraction pattern. The solid line is the diffraction pattern of the grease produced in Example 1, and the broken line is the diffraction pattern of the grease produced in Comparative Example 2.

Description of Embodiments

[(a) Thickener]

[0012] The thickener that can be used in the present invention contains a mixture of diurea compounds represented by the following formulas (I), (II), and (III):

(I): R1-NHCONH-R2-NHCONH-R1,

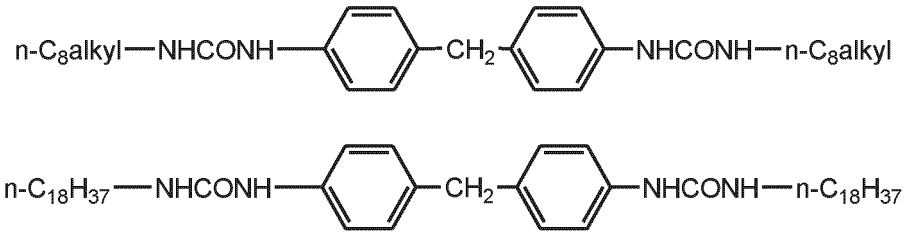
(II): R3-NHCONH-R2-NHCONH-R3,

and

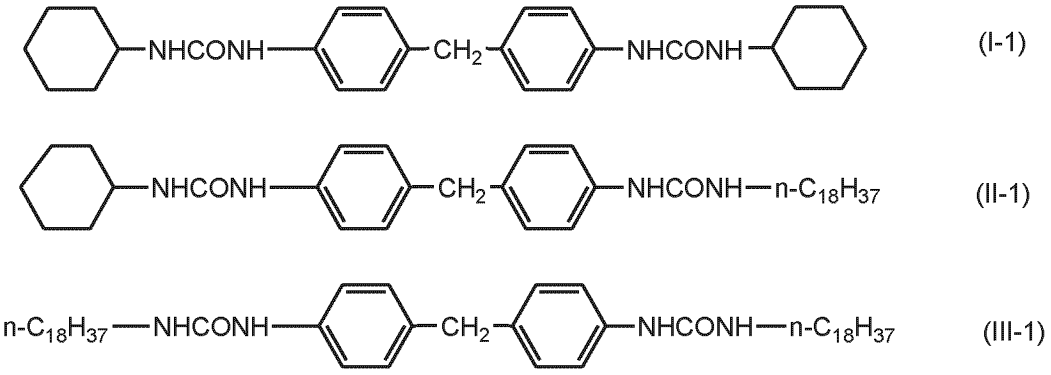
(III): R1-NHCONH-R2-NHCONH-R3.

[0013] In the formulas, R1 is a cyclohexyl group, R2 is a C6-15 divalent aromatic hydrocarbon group, R3 is a C8-20 linear or branched alkyl group or alkenyl group, and a ratio of the number of moles of the cyclohexyl group to the total number of moles of the cyclohexyl group and the alkyl group or alkenyl group [{R1/(R1+R3)}×100] (hereinafter sometimes referred to as "cyclohexyl%" or "CH%") is 90 to 0 mol%, preferably 80 to 0 mol%.

[0014] A thickener having a cyclohexyl% of 0 mol% is called an aliphatic diurea thickener and is synthesized from a diisocyanate such as diphenylmethane-4,4'-diisocyanate and a monoamine having a C8-20 linear or branched alkyl group or alkenyl group. As the aliphatic diurea, a compound synthesized from diphenylmethane-4,4'-diisocyanate and octylamine, octadecylamine, or a mixture thereof is preferable. The structural formulas of more preferable aliphatic diureas are shown below.



[0015] A thickener having a cyclohexyl% of more than 0 mol% and 90 mol% or less is called an alicyclic-aliphatic diurea and is synthesized from a diisocyanate such as diphenylmethane-4,4'-diisocyanate, cyclohexylamine, and a monoamine having a C8-20 linear or branched alkyl group or alkenyl group. Since alicyclic-aliphatic diurea is synthesized using two different amines, it is a mixture of an alicyclic diurea represented by formula (I), an aliphatic diurea represented by formula (II), and an alicyclic-aliphatic diurea represented by formula (III). A more preferable alicyclic-aliphatic diurea is a mixture of the following structural formulas (I-1) to (III-1).



[0016] As the thickener of the present invention, aliphatic diurea is preferable. Among them, aliphatic diurea synthesized from diphenylmethane-4,4'-diisocyanate and octylamine or octadecylamine is preferable. Aliphatic diurea synthesized from diphenylmethane-4,4'-diisocyanate and octylamine or octadecylamine is preferable from the viewpoints of shear stability and fluidity.

[0017] The content of the thickener in the grease composition of the present invention is not particularly limited, but is preferably 3 to 20 mass%, more preferably 6 to 18 mass%, and even more preferably 9 to 16 mass%, based on the total mass of the composition, from the viewpoints of fluidity and durability.

[(b) Base Oil]

[0018] The base oil usable in the present invention is not particularly limited. Mineral oil, synthetic oil, or a mixture thereof can be used. Examples of mineral oils include paraffinic mineral oils and naphthenic mineral oils. Examples of synthetic oils include ester-based synthetic oils such as diesters and polyol esters; synthetic hydrocarbon oils such as poly-α-olefin and polybutene; ether-based synthetic oils such as alkyldiphenyl ether and polypropylene glycol; silicone oils; and fluorinated oils. The synthetic oil may be a so-called biomass oil produced from biological resources as raw materials derived from animals and plants. For example, it is also possible to use biomass ester oils synthesized from various fatty acids and alcohols derived from vegetable oils as raw materials, and biomass hydrocarbon oils using vegetable oils such as palm oil, corn oil, and soybean oil. The base oil may be used alone or in admixture of two or more.

[0019] As the base oil of the present invention, mineral oil, poly-α-olefin, polyol ester, and alkyldiphenyl ether are preferable, and it is more preferable to contain mineral oil and poly-α-olefin. The mineral oil is preferably a paraffinic mineral oil, and is more preferably a Group II paraffinic mineral oil (i.e., sulfur content of 0.03 mass% or less, saturates of 90 vol% or more, viscosity index of 80 or more and less than 120) or a Group III paraffinic mineral oil (i.e., sulfur content of 0.03 mass% or less, saturates of 90 vol% or more, viscosity index of 120 or more) in the American Petroleum Institute classification.

[0020] When the base oil is mineral oil and/or poly-α-olefin and another base oil is further contained, the entirety preferably contains 50 mass% or more, more preferably 80 mass% or more, and even more preferably 90 mass% or more of mineral oil and/or poly-α-olefin based on the total mass of the base oil.

[0021] The kinematic viscosity of the base oil used in the present invention is not particularly limited, but the kinematic viscosity at 100°C is preferably 5 to 30 mm^2/s, more preferably 7 to 20 mm^2/s, and even more preferably 10 to 16 mm^2/s. When the kinematic viscosity of the base oil is in such a range, it is preferable from the viewpoints of durability and heat

generation.

[0022] The content of the base oil is preferably 50 to 95 mass%, more preferably 70 to 95 mass%, and even more preferably 80 to 90 mass%, based on the total mass of the composition. When the proportion of the base oil is in such a range, it is preferable from the viewpoint of fluidity.

[0023] The penetration is preferably 280 to 360, more preferably 300 to 350, and particularly preferably 300 to 330 from the viewpoint of handling properties. In this specification, the term "penetration" means 60-stroke worked penetration measured according to JIS K2220 7. In addition, unless otherwise specified, it refers to the penetration of unused grease.

[Shear Stability by Roll Stability Test]

[0024] The shear stability of grease can be evaluated using the penetration after the roll stability test as a criterion. Unused grease is used for the roll stability test.

[0025] From the viewpoint of preventing leakage and scattering from the lubricating portion and preventing churning, the penetration after the roll stability test is preferably 440 or less. It is more preferably 420 or less, and even more preferably 400 or less. It is particularly preferably 380 or less, and most preferably 360 or less.

[Shear Stability by X-ray Diffraction]

[0026] The shear stability of grease is determined by the structural stability of the thickener, and the structural stability of the thickener depends on the type of thickener and the state of the crystal. The crystal structure of the urea-based thickener has a stable type or a metastable type (a type that easily transitions to the stable type by applying energy), and when the thickener has a stable type structure, the grease exhibits good shear stability.

[0027] Whether the crystal structure of the urea thickener is stable or metastable can be determined by measuring the intermolecular distance (=molecular cell size) by XRD. In the metastable type, since the molecules are connected straight to form crystals, a peak is detected at a position of the molecular cell size close to the molecular size (maximum value) geometrically calculated in theory; however, in the stable type, since the molecules are connected obliquely to form crystals, a peak is detected at a position of a smaller molecular cell size away from the molecular size (maximum value) geometrically calculated in theory.

[0028] Therefore, in the present invention, as defined in the following formula (A), when the diurea thickener has a peak in which the value obtained by dividing the molecular cell size of the diurea thickener measured by X-ray diffraction by the molecular cell size of the diurea thickener represented by the same chemical formula obtained by geometric calculation is in the range of 0.7 to 0.4, it is determined that the crystal structure is in a stable form and the grease has better shear stability. The calculation was performed by the molecular mechanics method using MMFF94 (Merck Molecular Force Field 94) as the molecular force field. Specifically, the molecular shape in the single molecule state was determined using the energy optimization function of the molecular editor software "Avogadro," and the molecular size was calculated. The thickener to be measured by X-ray diffraction is a diurea thickener contained in the grease before use (that is, before being sheared at the lubrication portion).

[Molecular Cell Size of Diurea Thickener Measured by X-ray Diffraction] ÷ [Molecular Cell Size of the Diurea Thickener Obtained by Geometric Calculation] = 0.7 to 0.4 (A)

[0029] The urea-based grease to which the grease composition of the present invention belongs is generally produced through a step of reacting an isocyanate and an amine, and then a step of growing a urea crystal structure by heating and holding the reaction product. In the former reaction step, the molecular-level structure of the thickener is determined, and in the latter growth step, the crystal-level structure of the thickener is determined. After the growth step, the target grease is obtained through a step of dispersing the thickener. In the final dispersion step, a size more macro than the crystal level is controlled.

[0030] Generally, the reaction temperature in the reaction step is 80°C or lower, and the heating temperature in the growth step is 120 to 180°C; however, in the present invention, by setting the heating temperature in the growth step higher than usual and changing the subsequent holding time from the conventional one, a grease composition containing a thickener having a stable crystal structure was successfully obtained. The heating temperature is preferably higher than 190°C, more preferably 195°C or higher, preferably 220°C or lower, and more preferably 210°C or lower. The holding time after heating is preferably 1 hour or less, and more preferably 30 minutes or less, although it depends on the heating temperature. The holding time may be 0 hours.

[Additive]

[0031] The grease composition of the present invention can contain additives commonly used in various lubricating oils and greases. Commonly used additives include the following:

- Solid additives (e.g., molybdenum disulfide, graphite or melamine cyanurate (MCA), polytetrafluoroethylene (PTFE), metal oxide salts (e.g., CaO, ZnO, MgO), metal carbonate salts (e.g., $CaCO_3$, $ZnCO_3$), and metallic soaps),
- Extreme pressure agents (e.g., sulfide olefins, sulfide esters or sulfide oils and/or fats, sulfur-phosphorus-based compounds (e.g., triphenyl phosphorothionate),

    •• Organometallic extreme pressure agents (Zn, Mo, Sb, Bi, and other salts of dialkyl dithiophosphoric acid, Zn, Mo, Sb, Ni, Cu, Bi, and other salts of dialkyl dithiocarbamic acid, and others; ashless dithiocarbamates, ashless dithiophosphate carbamates, and the like)),

- Antiwear agents (e.g., phosphate esters, acidic phosphate esters, and acidic phosphate ester amine salts),
- Oiliness agents (e.g., triglycerides, diglycerides, monoglycerides, fatty acids, alcohols, amines, esters, and animal and vegetable oils and/or fats),
- Friction modifiers (e.g., polyethylene wax, oxidized polyethylene wax, polypropylene wax, montan wax, and amide wax),
- Rust inhibitors (e.g., fatty acid amine salts, zinc naphthenates or metal sulfonates, metal soaps, carboxylic acid partial esters of polyhydric alcohols, carboxylic acids and derivatives thereof (e.g., alkenyl succinic anhydrides, alkenyl succinic esters, and alkenyl succinic half esters), and esters (e.g., sorbitan trioleate and sorbitan monooleate)),
- Passivators (e.g., Na nitrite and Na molybdate)),
- Metal corrosion inhibitors (e.g., benzotriazoles or thiadiazoles, and zinc oxide),
- Metal-based detergents (e.g., alkaline earth metal sulfonates, alkaline earth metal phenates, and alkaline earth metal salicylates),
- Ashless dispersants (e.g., polybutenyl succinimide-based, polybutenyl succinamide-based, benzylamine-based, and succinic acid ester-based),
- Antioxidants (e.g., amine-based antioxidants and phenol-based antioxidants).

    •• Amine-based antioxidants (reaction products of N-phenylbenzylamine and 2,4,4-trimethylpentene, alkyldiphenylamines such as octylated diphenylamine, N-n-butyl-p-aminophenol, 4,4'-tetramethyl-di-aminodiphenylmethane, α-naphthylamine, N-phenyl-α-naphthylamine, and phenothiazine),
    •• Phenol-based antioxidants (e.g., 2,6-di-tert-butyl-p-cresol (BHT), 2,2'-methylenebis(4-methyl-6-tert-butylphenol), 4,4'-butylidenebis(3-methyl-6-tert-butylphenol), 2,6-di-tert-butyl-phenol, 2,4-dimethyl-6-tert-butylphenol, tert-butylhydroxyanisole (BHA), 4,4'-butylidenebis(3-methyl-6-tert-butylphenol), 4,4'-methylenebis(2,3-di-tert-butylphenol), and 4,4'-thiobis(3-methyl-6-tert-butylphenol)),

- Viscosity index improvers (e.g., polymethacrylate-based, polyisobutylene-based, ethylene-propylene copolymer-based, olefin copolymer-based, and styrene-butadiene hydrogenated copolymer-based),
- Pour point depressants (e.g., ethylene-vinyl acetate copolymers, condensates of chlorinated paraffin and naphthalene, condensates of chlorinated paraffin and phenol, polymethacrylates, and polyalkylstyrenes).

[Applications]

[0032] The grease composition of the present invention can be used, for example, for machine parts having steel lubricated portions that perform rolling motion and rolling/sliding motion, and typical examples thereof include rolling bearings, gears, ball screws, linear guide bearings, joints, and cams.

[0033] Specifically, it can be used for rolling bearings used in various motors for industrial machinery, various motors for office equipment, and various motors for automobiles; rolling bearings used in automotive electrical components and auxiliary components such as automotive wheel bearings, alternators, electromagnetic clutches, idler pulleys, and timing belt tensioners; gears used in reducers and speed increasers of wind turbines, robots, and automobiles; ball screws used in electric power steering and machine tools; linear guide bearings used in industrial equipment and electronic equipment; and constant velocity joints used in automotive drive shafts and propeller shafts. It is particularly suitable for use in reducers, speed increasers, drive shafts, and propeller shafts.

Examples

[Grease Preparation]

[0034] A base oil and 1 mole of diphenylmethane-4,4'-diisocyanate were placed in a vessel, and the mixture was heated to 70 to 80°C. A base oil and 2 moles of amine were placed in a separate vessel, heated to 70 to 80°C, and then added to the previous vessel, followed by reaction for 30 minutes with good stirring. Then, the temperature was raised to the temperature described in Tables 1 and 2 with stirring, held for about 30 minutes, and then allowed to cool to obtain a base grease. To this base grease, an amine-based antioxidant was added in an amount of 1 mass% based on the total mass of the finally obtained grease, a base oil was added as appropriate, and the resulting mixture was adjusted to a penetration of 300 using a three-roll mill.

[0035] The raw materials used for the preparation of the grease are as follows:

<Base Oil>
Mineral oil: Kinematic viscosity at 40°C: 12 mm^2/s, measured according to JIS K2220 83
<Amine>

Raw material amine for "aliphatic diurea (C8)": Octylamine
Raw material amine for "aliphatic diurea (C18)": Octadecylamine
Raw material amine for "aliphatic diurea (C8/C18)": Octylamine: Octadecylamine=5:5 (molar ratio)
Amines constituting "alicyclic-aliphatic diurea": Octadecylamine: Cyclohexylamine=9:1 (molar ratio)

<Additive>
Amine-based antioxidant: Irganox L-57

[Evaluation of Shear Stability by Roll Stability Test]

[0036] The shear stability of the grease compositions of Examples and Comparative Examples obtained above was evaluated by a roll stability test. The roll stability was measured by the method specified in ASTM D 1831. However, the temperature and test time were changed to 120°C and 24 hours. The results are shown in Tables 1 and 2.

◦: 440 or less
×: Greater than 440

[0037] The penetration used as the criterion is the penetration after 60 strokes of working after the roll stability test.

[Evaluation of Shear Stability by X-ray Diffraction]

[0038] The shear stability of the grease compositions of Examples and Comparative Examples obtained above was evaluated by X-ray diffraction. An X-ray diffractometer (XRD, D8 DISCOVER, manufactured by Bruker) was used for the measurement. The measurement conditions were as follows: target: Cu, tube voltage: 40 kV, tube current: 40 mA, sampling width: 2θ=0.015°, scan speed: 2θ=1.5°/min. The calculation was performed by the molecular mechanics method using MMFF94 as the molecular force field, as described above.

[0039] The grease compositions were evaluated by the presence or absence of a peak corresponding to the above formula (A) among peaks measured by X-ray diffraction. The results are shown in Tables 1 and 2. Further, the X-ray diffraction patterns of Example 1 and Comparative Example 2 are shown in Figure 1.

Table 1

	Example 1	Example 2	Example 3	Example 4
Thickener	Aliphatic Diurea (C18)	Aliphatic Diurea (C18)	Aliphatic Diurea (C8)	Alicyclic-Aliphatic Diurea
Base Oil	Mineral Oil	Mineral Oil	Mineral Oil	Mineral Oil
Base Grease Heating Condition	195°C	200°C	170°C	170°C
Shear Stability	○ 328	○ 362	○ 349	○ 385

(continued)

	Example 1	Example 2	Example 3	Example 4
X-ray Diffraction	Present, Approx. 0.63	Present, Approx. 0.63	-	-

Table 2

	Comparative Example 1	Comparative Example 2	Comparative Example 3
Thickener	Aliphatic Diurea (C8/C18)	Aliphatic Diurea (C18)	Aliphatic Diurea (C18)
Base Oil	Mineral Oil	Mineral Oil	Mineral Oil
Base Grease Heating Condition	170°C	170°C	190°C
Shear Stability	× 440 <	× 440 <	× 440 <
X-ray Diffraction	-	Absent, Approx. 0.77	Absent, Approx. 0.77

Claims

1. A grease composition comprising

(a) a thickener containing a mixture of diurea compounds represented by the following formulas (I), (II), and (III):

(I): R1-NHCONH-R2-NHCONH-R1,

(II): R3-NHCONH-R2-NHCONH-R3,

and

(III): R1-NHCONH-R2-NHCONH-R3,

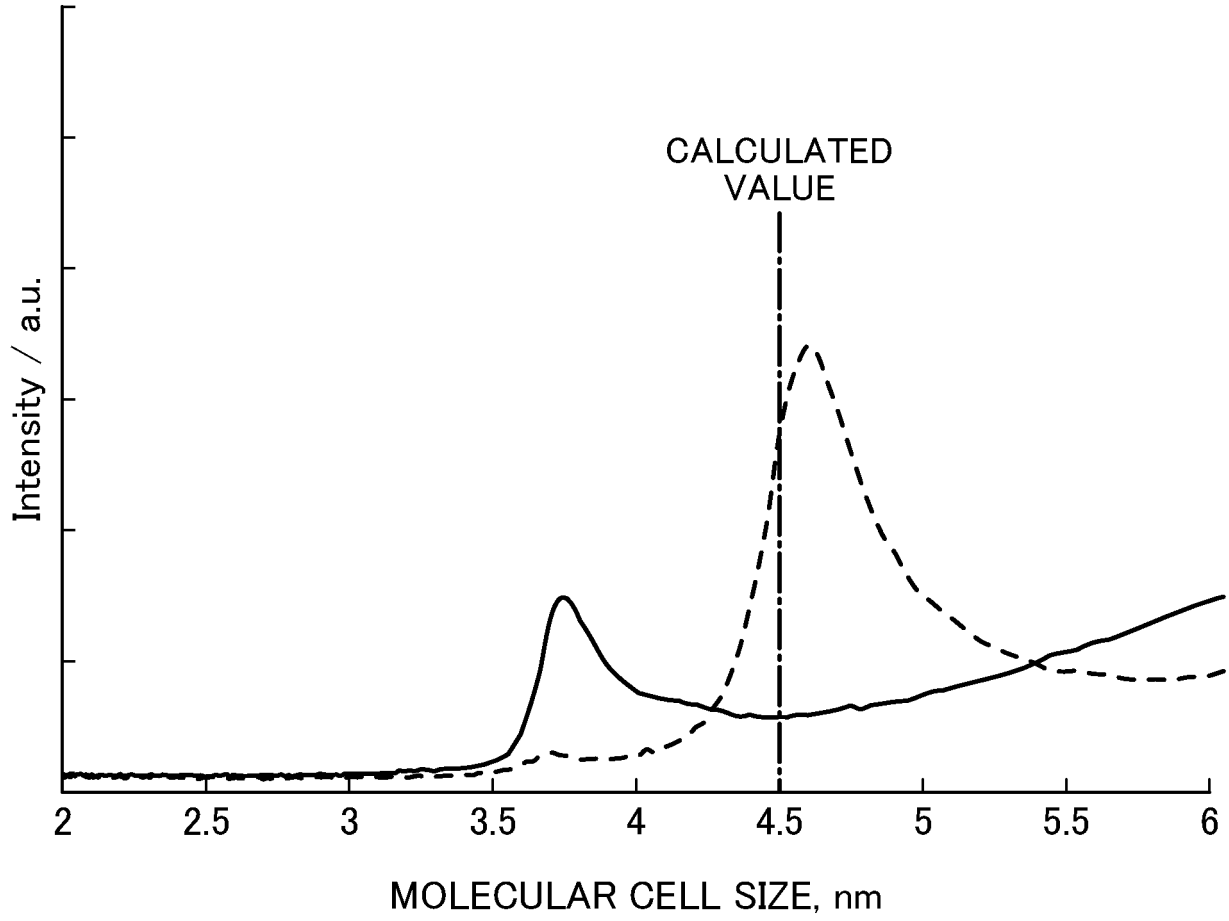
where R1 is a cyclohexyl group, R2 is a C6-15 divalent aromatic hydrocarbon group, R3 is a C8-20 linear or branched alkyl group or alkenyl group, and a ratio of the number of moles of the cyclohexyl group to the total number of moles of the cyclohexyl group and the alkyl group or alkenyl group [{R1/(R1+R3)}×100] is 80 to 0 mol%, and
(b) a base oil,

wherein a 60-stroke worked penetration measured by a method specified in JIS K 2220.7 is 300 to 350, and a penetration in a roll stability test measured after applying shearing at 120°C for 24 hours using a tester conforming to ASTM D 1831 is 440 or less.

2. The grease composition according to claim 1, wherein the diurea thickener exhibits a peak corresponding to the following formula (A) among peaks measured by X-ray diffraction:

[Molecular Cell Size of Diurea Thickener Measured by X-ray Diffraction] ÷ [Molecular Cell Size of the Diurea Thickener Obtained by Geometric Calculation] = 0.7 to 0.4 (A)

FIG.1


CALCULATED
VALUE
Intensity / a.u.
2
2.5
3
3.5
4
4.5
5
5.5
6
MOLECULAR CELL SIZE, nm

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2023/014691

A. CLASSIFICATION OF SUBJECT MATTER

C10N 20/00(2006.01)n; *C10N 30/00*(2006.01)n; *C10N 30/06*(2006.01)n; *C10N 40/02*(2006.01)n; *C10N 40/04*(2006.01)n; *C10N 50/10*(2006.01)n; ***C10M 115/08***(2006.01)i
FI: C10M115/08; C10N20:00 Z; C10N30:00 Z; C10N40:02; C10N40:04; C10N50:10; C10N20:00 B; C10N30:06

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)

C10N20/00; C10N30/00; C10N30/06; C10N40/02; C10N40/04; C10N50/10; C10M115/08

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	JP 2015-160909 A (SHOWA SHELL SEKIYU K.K.) 07 September 2015 (2015-09-07) paragraph [0041], example 20	1-2
X	JP 06-017080 A (SHOWA SHELL SEKIYU K.K.) 25 January 1994 (1994-01-25) example 8, comparative example 2	1-2
X	JP 09-324190 A (KYODO YUSHI CO., LTD.) 16 December 1997 (1997-12-16) examples 1-10, 13-15, comparative examples 1-6	1-2
X	JP 03-119097 A (SUMITOMO ELECTRIC INDUSTRIES, LTD.) 21 May 1991 (1991-05-21) examples 1, 8, comparative example 2	1-2
X	JP 01-268793 A (KYODO YUSHI CO., LTD.) 26 October 1989 (1989-10-26) comparative examples 2, 3, 5	1-2
X	JP 2010-144042 A (SHOWA SHELL SEKIYU K.K.) 01 July 2010 (2010-07-01) comparative example 2	1-2

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

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
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15 June 2023	**27 June 2023**
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2023/014691

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	JP 09-104889 A (THE LUBRIZOL CORP.) 22 April 1997 (1997-04-22) example 1	1-2
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