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(54) **GREASE COMPOSITION**

(57) The present invention addresses a problem of providing a grease composition for a speed reducer and a speed increaser, excellent in both leakage prevention performance and energy transfer efficiency. The grease composition for a speed reducer and a speed increaser

contains a base oil (A), and nanofibers (B) having a thickness (d) of 1 to 500 nm, wherein the nanofibers (B) are one or more selected from cellulose nanofibers (B1) and modified cellulose nanofibers (B2).

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Description

Technical Field

5 **[0001]** The present invention relates to a grease composition. More specifically, the present invention relates to a grease composition used in a speed reducer and a speed increaser.

Background Art

10 **[0002]** Grease is easier to seal than lubricating oil, and can reduce the size and weight of the machine to which it is applied. Therefore, it has been widely used for lubrication of various sliding parts of automobiles, electric appliances, industrial machinery, industrial machines and the like.

[0003] In recent years, grease has also been used in speed reducers used in industrial robots and the like and speed increasers used in wind power generation facilities and the like (see, for example, PTL 1).

15 **[0004]** The speed reducer has a mechanism that applies torque to the input side to reduce the speed and transmit the torque to the output side.

[0005] The speed increaser has a mechanism that applies torque to the input side to increase the speed and transmit the torque to the output side.

20 **[0006]** Grease used in lubricating portions of a speed reducer and a speed increaser is required to have excellent energy transfer efficiency from the viewpoint of suppressing loss of torque (energy) applied to the input side and transmitting the torque to the output side without waste.

Citation List

25 Patent Literature

[0007] PTL 1: JP 2017-203069 A

Summary of Invention

30 **[0008]** In the case of preparing a grease excellent in energy transfer efficiency, it is general to increase the worked penetration to obtain a soft grease. However, when a soft grease having a high worked penetration is used, there is a problem that the grease leakage prevention performance is deteriorated.

[0009] When leakage of grease occurs due to deterioration of the grease leakage prevention performance, the grease adheres to a product manufactured by an apparatus including a speed reducer or an apparatus including a speed increaser, and the like, resulting in deterioration of the product yield. In addition, from the viewpoint of safety and the like, foods produced by food production apparatuses and the like are strongly required to prevent foreign substances from being mixed into the foods. From such a viewpoint, prevention of adhesion of grease to the food is strongly demanded.

40 **[0010]** Therefore, although it is desired to improve the grease leakage prevention performance, there has been a problem that the grease leakage prevention performance cannot be sufficiently ensured when the worked penetration of the grease is increased in order to improve the energy transfer efficiency.

45 **[0011]** The object of the present invention is to provide a grease composition for a speed reducer and a speed increaser, which is excellent in both leakage prevention performance and energy transfer efficiency.

Solution to Problem

50 **[0012]** The present inventors have found that a grease composition containing a base oil and a specific nanofiber can solve the aforementioned problems, and have completed the present invention.

[0013] Specifically, the present invention is concerned with the following [1] to [10].

[1] A grease composition used for a speed reducer and a speed increaser, containing: a base oil (A); and nanofibers (B) having a thickness (d) of 1 to 500 nm, wherein the nanofibers (B) are one or more selected from cellulose nanofibers (B1) and modified cellulose nanofibers (B2).

[2] The grease composition as set forth in [1], wherein the content of the nanofibers (B) is 0.1 to 20% by mass based on the total amount of the grease composition.

[3] The grease composition as set forth in [1] or [2], further containing an organic bentonite (C), wherein the nanofibers

(B) contain the cellulose nanofibers (B1).

[4] The grease composition as set forth in [3], wherein the content of the organic bentonite (C) is 0.01 to 15% by mass based on the total amount of the grease composition.

[5] The grease composition as set forth in [3] or [4], wherein a content ratio [B1/C] of the cellulose nanofibers (B1) to the organic bentonite (C) is 0.05 to 5.0 in terms of mass ratio.

[6] The grease composition as set forth in any of [1] to [5], wherein the base oil (A) is a mixed base oil containing a low-viscosity base oil (A1) having a kinematic viscosity at 40°C of 5 to 150 mm²/s and a high-viscosity base oil (A2) having a kinematic viscosity at 40°C of 200 to 1000 mm²/s.

[7] The grease composition as set forth in any of [1] to [6], wherein the grease composition has a worked penetration at 25°C of 220 to 440.

[8] The grease composition as set forth in any of [1] to [7], wherein the grease composition is used in a food machine including a speed reducer or a food machine including a speed increaser.

[9] A lubrication method, including lubricating a lubricating portion of a speed reducer or a speed increaser with the grease composition as set forth in any of [1] to [7].

[10] A lubrication method, including lubricating a lubricating portion of a food machine including a speed reducer or a speed increaser with the grease composition as set forth in any of [1] to [7].

Advantageous effects of Invention

[0014] According to the present invention, it is possible to provide a grease composition for a speed reducer and a speed increaser, which is excellent in both leakage prevention performance and energy transfer efficiency.

Brief Description of Drawings

[0015] Fig. 1 is a schematic view of an apparatus used for measuring torque transmission efficiency as an index of energy transfer efficiency in this embodiment.

Description of Embodiments

[0016] In the description herein, with respect to a preferable numerical range (for example, a range of content or the like), the lower limit value and the upper limit value described in stages can be independently combined with each other. For example, from the description of "preferably 10 to 90, and more preferably 30 to 60", "preferable lower limit value (10)" and "more preferable upper limit value (60)" can be combined to form "10 to 60".

[0017] In addition, in the description herein, the numerical values in Examples are numerical values that can be used as upper limit values or lower limit values.

[Embodiment of Grease Composition of the Present Invention]

[0018] The grease composition of the present invention is a grease composition used for a speed reducer and a speed increaser, containing a base oil (A), and nanofibers (B) having a thickness (d) of 1 to 500 nm, wherein the nanofibers (B) are one or more selected from cellulose nanofibers (B1) and modified cellulose nanofibers (B2).

[0019] In the grease composition of the present invention, the thickness (d) of the nanofibers (B) contained in the grease composition is defined. In other words, the thickness (d) of the nanofibers (B) dispersed in the base oil (A) is defined.

[0020] Satisfying the definition, the nanofibers (B) can readily form a higher-order structure in the base oil (A). In addition, the nanofibers (B) are easily uniformly dispersed in the base oil (A). As a result, even if the content of the nanofibers (B) is small, it is possible to obtain a grease composition that can be easily adjusted to an appropriate worked penetration and is excellent in both leakage prevention performance and energy transfer efficiency.

[0021] In the description herein, in the following description, "base oil (A)" and "nanofibers (B)" are also referred to as "component (A)" and "component (B)", respectively. Also, "cellulose nanofibers (B1)" and "modified cellulose nanofibers (B2)" are also referred to as "component (B1)" and "component (B2)", respectively.

[0022] In the lubricating oil composition according to one embodiment of the present invention, the total content of the component (A) and the component (B) is preferably 50% by mass or more, more preferably 60% by mass or more, still more preferably 70% by mass or more, and yet still more preferably 80% by mass or more, based on the total amount (100% by mass) of the grease composition.

[0023] In the grease composition according to one embodiment of the present invention, the upper limit value of the total content of the component (A) and the component (B) may be adjusted in relation to the content of additives other than the component (B), and is preferably 99% by mass or less, more preferably 95% by mass or less, and still more preferably 92% by mass or less.

[0024] Hereinafter, the base oil (A) and the nanofibers (B) will be described in detail.

<Base Oil (A)>

[0025] The base oil (A) contained in the grease composition of the present invention is not particularly limited, and examples thereof include mineral oil, synthetic oil, animal oil, vegetable oil, and liquid paraffin.

[0026] The base oil (A) may be either a base oil composed of a single kind or a mixed base oil of two or more kinds thereof.

(Mineral Oil)

[0027] Examples of the mineral oil include distillate oils obtained through atmospheric distillation of paraffin crude oils, intermediate base crude oils or naphthenic crude oils, or through vacuum distillation of atmospheric distillation residues; refined oils obtained by subjecting such distillate oils to at least one or more refining treatments selected from refining treatments such as solvent deasphalting, solvent extraction, hydrocracking or hydrotreating, as well as refining treatments such as solvent dewaxing or catalytic dewaxing (specifically, a solvent-refined oil, a hydrogenated refined oil, a dewaxing treated oil, a white clay treated oil).

[0028] Among those mineral oils, mineral oils classified into Group 3 of the base oil category according to API (American Petroleum Institute) are preferred.

(Synthetic Oil)

[0029] Examples of the synthetic oil include hydrocarbon-based oils, aromatic oils, ester-based oils, ether-based oils, and fatty acid esters.

[0030] Examples of the hydrocarbon-based oil include a normal paraffin, an isoparaffin, a poly- α -olefin (PAO), such as polybutene, polyisobutylene, a 1-decene oligomer, a co-oligomer of 1-decene and ethylene, and hydrides thereof. In addition, a GTL synthetic oil obtained by isomerizing a wax (GTL wax (Gas To Liquids WAX)) produced by the Fischer-Tropsch process or the like is also included.

[0031] Examples of the aromatic oil include alkylbenzenes, such as a monoalkylbenzene and a dialkylbenzene; and alkylnaphthalenes, such as a monoalkylnaphthalene, a dialkylnaphthalene, and a polyalkylnaphthalenes.

[0032] Examples of the ester-based oil include diester-based oils, such as dibutyl sebacate, di-2-ethylhexyl sebacate, dioctyl adipate, diisodecyl adipate, ditridecyl adipate, ditridecyl glutarate, and methyl acetyl ricinoleate; aromatic ester-based oils, such as trioctyl trimellitate, tridecyl trimellitate, and tetraoctyl pyromellitate; polyol ester-based oils, such as trimethylolpropane caprylate, trimethylolpropane pelargonate, pentaerythritol-2-ethyl hexanoate, and pentaerythritol pelargonate; and complex ester-based oils, such as an oligo ester between a polyhydric alcohol and a mixed fatty acid of a dibasic acid and a monobasic acid.

[0033] Examples of the ether-based oil include polyglycols, such as polyethylene glycol, polypropylene glycol, polyethylene glycol monoether, and polypropylene glycol monoether; and phenyl ether-based oils, such as a monoalkyl triphenyl ether, an alkyl diphenyl ether, a dialkyl diphenyl ether, pentaphenyl ether, tetraphenyl ether, a monoalkyl tetraphenyl ether, and a dialkyl tetraphenyl ether.

[0034] The fatty acid that constitutes the fatty acid ester is preferably a fatty acid having 8 to 22 carbon atoms, and specifically, examples thereof include caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, behenic acid, erucic acid, palmitoleic acid, oleic acid, linoleic acid, linolenic acid, isostearic acid, arachidic acid, ricinoleic acid, and 12-hydroxystearic acid.

[0035] Specifically, examples of the fatty acid ester include a glycerin fatty acid ester, a polyglycerin fatty acid ester, and a propylene glycol fatty acid ester.

[0036] Examples of the glycerin fatty acid ester include glycerin monooleate, glycerin monostearate, glycerin monocaprylate, glycerin dioleate, glycerin distearate, and glycerin dicaprylate.

[0037] Examples of the polyglycerin fatty acid ester include diglycerin monooleate, diglycerin monoisostearate, diglycerin dioleate, diglycerin trioleate, diglycerin monostearate, diglycerin distearate, diglycerin tristearate, diglycerin triisostearate, diglycerin monocaprylate, diglycerin dicaprylate, diglycerin tricaprylate, triglycerin monooleate, triglycerin dioleate, triglycerin trioleate, triglycerin tetraoleate, triglycerin monostearate, triglycerin distearate, triglycerin tristearate, triglycerin tetrastearate, triglycerin monocaprylate, triglycerin dicaprylate, triglycerin tricaprylate, triglycerin tetracaprylate, diglycerin monooleic acid monostearic acid ester, diglycerin monooleic acid distearic acid ester, diglycerin monocaprylic acid monostearic acid ester, triglycerin monooleic acid monostearic acid ester, triglycerin dioleic acid distearic acid ester, triglycerin dioleic acid monostearic acid ester, triglycerin monooleic acid monostearic monocaprylic acid ester, diglycerin monolaurate, diglycerin dilaurate, triglycerin monolaurate, triglycerin trilaurate, triglycerin trilaurylate, diglycerin monomyristate, diglycerin dimyristate, triglycerin monomyristate, triglycerin dimyristate, triglycerin trimyristate, diglycerin monolinoleate, diglycerin dilinoleate, triglycerin monolinoleate, triglycerin dilinoleate, triglycerin trilinoleate, decaglycerin

monooleate, decaglycerin monostearate, and decaglycerin monocaprylic acid monooleic acid ester.

[0038] Examples of the propylene glycol fatty acid ester include propylene glycol monooleate, propylene glycol monostearate, propylene glycol monocaprylate, and propylene glycol monolaurate.

(Vegetable Oil)

[0039] The vegetable oil is a plant-derived oil, and specifically, examples thereof include rapeseed oil, peanut oil, corn oil, cottonseed oil, canola oil, soybean oil, sunflower oil, palm oil, coconut oil, safflower oil, camellia oil, olive oil, and groundnut oil.

(Animal Oil)

[0040] The animal oil is an animal-derived oil, and specifically, examples thereof include lard, neat's foot oil, chrysalis oil, sardine oil, and herring oil.

(Liquid Paraffin)

[0041] Examples of the liquid paraffin include alicyclic hydrocarbon compounds having a branched structure or a ring structure and represented by C_mH_n (m is number of carbon atoms, provided that $n < (2m + 2)$), and mixtures thereof.

[0042] Among the aforementioned base oils, from the viewpoint of affinity with the nanofibers (B), the base oil (A) contained in the grease composition according to one embodiment of the present invention preferably contains one or more selected from mineral oils classified into Group 3 of the base oil category according to API, synthetic oils, vegetable oils, animal oils, fatty acid esters, and liquid paraffins, and more preferably one or more selected from mineral oils classified into Group 3 of the base oil category according to API and synthetic oils. As the synthetic oil, poly- α -olefin (PAO) is preferably used.

[0043] Here, in the case where the grease composition is required to have oxidation stability at a high temperature, a synthetic oil is preferably used, one or more selected from a hydrocarbon-based oil, an ester-based oil, and an ether-based oil are more preferably used, and a hydrocarbon-based oil is still more preferably used. In addition, by using a mixture of a hydrocarbon-based oil, an ester-based oil, and an ether-based oil, heat resistance, sealing resistance, and low-temperature characteristics can be balanced, and from this viewpoint, a hydrocarbon-based oil is preferably used.

(Kinematic Viscosity and Viscosity Index of Base Oil (A))

[0044] The base oil (A) used in one embodiment of the present invention has a kinematic viscosity at 40°C (hereinafter also referred to as "40°C kinematic viscosity") of preferably 10 to 400 mm²/s, more preferably 15 to 300 mm²/s, still more preferably 20 to 200 mm²/s, and yet still more preferably 20 to 130 mm²/s.

[0045] By adjusting the 40°C kinematic viscosity of the base oil (A) within the aforementioned range, the grease leakage prevention performance and energy transfer efficiency are easily improved.

[0046] The base oil (A) used in one embodiment of the present invention may be a mixed base oil obtained by combining a low-viscosity base oil (A1) and a high-viscosity base oil (A2) and adjusting the kinematic viscosity to the aforementioned range.

[0047] The low-viscosity base oil (A1) has the 40°C kinematic viscosity of preferably 5 to 150 mm²/s, more preferably 7 to 120 mm²/s, and still more preferably 10 to 100 mm²/s.

[0048] The high-viscosity base oil (A2) has the 40°C kinematic viscosity of preferably 200 to 1000 mm²/s, more preferably 250 to 800 mm²/s, and still more preferably 300 to 600 mm²/s.

[0049] A viscosity index of the base oil (A) that is used in one embodiment of the present invention is preferably 60 or more, more preferably 70 or more, and still more preferably 80 or more.

[0050] In the present invention, the 40°C kinematic viscosity and the viscosity index mean values as measured or calculated in conformity with JIS K2283:2000.

(Content of Base Oil (A))

[0051] The content of the base oil (A) contained in the grease composition according to one embodiment of the present invention is preferably 50% by mass or more, more preferably 60% by mass or more, still more preferably 70% by mass or more, and yet still more preferably 80% by mass or more, based on the total amount (100% by mass) of the grease composition.

<Nanofibers (B)>

[0052] The nanofibers (B) contained in the grease composition of the present invention are one or more types selected from cellulose nanofibers (B1) and modified cellulose nanofibers (B2).

[0053] When the grease composition contains the nanofibers (B), the nanofibers (B) are uniformly dispersed in the grease composition to form a higher-order structure. Since the nanofibers (B) have excellent mechanical stability, the higher-order structure of the nanofibers (B) is stable against shearing. Therefore, the shear stability of the grease composition is improved, and the grease leakage prevention performance is improved.

[0054] In addition, even when the content of the nanofibers (B) is small, the worked penetration of the grease composition can be adjusted to an appropriate range. Therefore, the proportion of the base oil (A) in the grease composition can be increased. Thus, the lubricity of the grease composition is enhanced, and the energy transfer efficiency is also easily improved.

(Cellulose Nanofibers (B1))

[0055] The cellulose nanofibers mean a fibrous material having a thickness of 500 nm or less, which is produced by defibrating plant fibers to a nano level, and is distinguished from a flaky material, a powdery material, and a granular material.

[0056] As a raw material for cellulose nanofibers, lignocellulose is also usable. It is known that lignocellulose is a composite hydrocarbon polymer that constitutes a cell wall of plants, and is mainly composed of polysaccharides of cellulose and hemicellulose and an aromatic polymer of lignin. The cellulose that constitutes cellulose nanofibers may be one or more selected from lignocellulose and acetylated lignocellulose. Cellulose nanofibers may contain one or more selected from hemicellulose and lignin. Further, the cellulose to constitute cellulose nanofibers may chemically bond to one or more selected from hemicellulose and lignin.

[0057] The degree of polymerization of cellulose constituting the cellulose nanofibers is preferably 50 to 3000, more preferably 100 to 1500, still more preferably 150 to 1000, and yet still more preferably 200 to 800.

[0058] In the description herein, the degree of polymerization of cellulose means a value as measured by a viscosity method.

(Modified Cellulose Nanofibers (B2))

[0059] The modified cellulose nanofibers are obtained by subjecting cellulose nanofibers to a modification treatment.

[0060] Specific examples of the modification treatment include esterification such as acetylation, phosphorylation, urethanization, carbamidation, etherification, carboxymethylation, TEMPO (2,2,6,6-tetramethylpiperidin-1-oxyl radical) oxidation, and periodate oxidation.

[0061] The modified cellulose nanofibers used in the present invention may be subjected to only one of these modification treatments, or may be subjected to two or more of these modification treatments.

[0062] In addition, resin-reinforced fibers containing one or more types selected from cellulose nanofibers and modified cellulose nanofibers and a thermoplastic resin are known. Such resin-reinforced fibers are also included in the modified cellulose nanofibers.

[0063] A thermoplastic resin and one or more types selected from the cellulose nanofibers and the modified cellulose nanofibers may be mixed or kneaded, or may be dispersed in each other.

[0064] Examples of the thermoplastic resin include polyethylene, polypropylene, polyvinyl chloride, polystyrene, polyvinylidene chloride, fluoro resin, (meth)acrylic resin, polyamide resin, polyester, polylactic acid resin, polylactic acid/polyester copolymer resin, acrylonitrile-butadiene-styrene copolymer, polycarbonate, polyphenylene oxide, (thermoplastic) polyurethane, polyacetal, vinyl ether resin, polysulfone resin, and cellulose resin (e.g., triacetylated cellulose, diacetylated cellulose). Here, (meth)acryl means acryl and/or methacryl.

[0065] The thermoplastic resin may be used alone or in combination of two or more kinds thereof.

("Thickness" of Nanofibers (B))

[0066] The definition of the "thickness" of the nanofibers (B) is the same as the definition relating to the thickness of a general fibrous material.

[0067] Specifically, in a cut surface at the time of cutting perpendicularly to the tangent direction in an arbitrary point on the side surface of the nanofibers (B), when the cut surface is a circle or an oval, the diameter or major axis is the "thickness" of the nanofibers (B). When the cut surface is a polygon, the diameter of the circumscribed circle of the polygon is the "thickness" of the nanofibers (B).

[0068] When the base oil (A) is blended with a flaky substance, a powdery substance or a granular substance having

a size of several pm or more (hereinafter also referred to as "micro-size particles") as a thickener, the micro-size particles tend to aggregate in the base oil (A) to form a so-called "lump". As a result, an aggregate of the micro-size particles is deposited on the surface of the obtained grease composition, and the dispersed state is liable to become non-uniform. In this case, it is necessary to add a large amount of the micro-size particles in order to increase the worked penetration of the obtained grease composition. However, as containing particles larger than the oil film thickness, the resultant grease composition becomes inferior in wear resistance.

[0069] On the other hand, in the grease composition of the present invention, since the nanofibers (B) having a thickness (d) of 1 to 500 nm are blended in the base oil (A), the nanofibers (B) do not aggregate in the base oil (A), and a higher-order structure is formed by the nanofibers (B) while the nanofibers (B) are uniformly dispersed. As a result, although the content of the nanofibers (B) is small, a grease composition having an appropriate worked penetration can be obtained.

(Thickness (d) and Aspect Ratio of Nanofibers (B))

[0070] In the present invention, the "thickness (d) of the nanofibers (B)" indicates the thickness of the nanofibers (B) dispersed in the base oil (A), and is distinguished from the "thickness (d') of the nanofibers (B)" as a raw material prior to being blended in the base oil (A).

[0071] However, there is almost no difference between the "thickness (d) of the nanofibers (B)" dispersed in the base oil (A) and the "thickness (d') of the nanofibers (B)" as a raw material prior to being blended in the base oil (A). Therefore, the "thickness (d) of the nanofibers (B)" dispersed in the base oil (A) and the "thickness (d') of the nanofibers (B)" as a raw material prior to being blended in the base oil (A) can be considered to be substantially the same.

[0072] The thickness (d) of the nanofibers (B) dispersed in the base oil (A) is 1 to 500 nm, but is preferably 1 to 300 nm, more preferably 1 to 200 nm, and still more preferably 2 to 100 nm from the viewpoint of forming a higher-order structure by the nanofibers (B) and from the viewpoint of more uniformly dispersing the nanofibers (B) in the base oil (A).

[0073] With regard to the nanofibers (B) contained in the grease composition of the present invention, it is sufficient to confirm dispersion of the nanofibers (B) having a thickness (d) within the aforementioned range, and the nanofibers (B) having a thickness (d) outside the aforementioned range may be dispersed.

[0074] However, in the grease composition according to one embodiment of the present invention, from the viewpoint of forming a higher-order structure by the nanofibers (B) and from the viewpoint of more uniformly dispersing the nanofibers (B) in the base oil (A), the average value of the thickness (d) of ten nanofibers (B) arbitrarily selected from the nanofibers (B) dispersed in the base oil (A) is preferably 1 to 500 nm, more preferably 1 to 300 nm, still more preferably 1 to 200 nm, and yet still more preferably 2 to 100 nm.

[0075] Also from the aforementioned viewpoint, among the nanofibers (B) contained in the grease composition of the present invention, in ten arbitrarily selected nanofibers, the number of the nanofibers (B) whose thickness (d) falls within the aforementioned range is preferably 1 or more, more preferably 5 or more, and still more preferably 7 or more. It is more preferred that all of the ten selected nanofibers (B) are the nanofibers (B) having a thickness (d) falling within the aforementioned range.

[0076] In the grease composition according to one embodiment of the present invention, an aspect ratio of the nanofibers (B) is preferably 5 or more, more preferably 10 or more, still more preferably 15 or more, yet still more preferably 30 or more, even more preferably 50 or more, yet even more preferably 70 or more, further more preferably 90 or more, and yet further more preferably 100 or more.

[0077] In the description herein, the "aspect ratio" refers to a ratio of a length to a thickness (length/thickness) of the nanofiber (B) to be observed, and the "length" of the nanofiber (B) refers to a distance between the two most distant points of the nanofiber (B).

[0078] In addition, when a part of the nanofiber (B) to be observed comes into contact with another nanofiber (B) and it is difficult to identify the "length", the length of only a part of the nanofiber (B) to be observed in which the thickness can be measured may be measured and the aspect ratio of the part may be in the aforementioned range.

[0079] Of the nanofibers (B) contained in the grease composition of the present invention, an average value of the aspect ratio (hereinafter also referred to as "average aspect ratio") of ten arbitrarily selected nanofibers (B) is preferably 5 or more, more preferably 10 or more, still more preferably 15 or more, yet still more preferably 30 or more, even more preferably 50 or more, yet even more preferably 70 or more, further more preferably 90 or more, and yet further more preferably 100 or more.

(Thickness (d') and Aspect Ratio of Nanofibers (B))

[0080] The thickness (d') of the nanofibers (B) as a raw material prior to being blended in the base oil (A) is preferably 1 to 500 nm, more preferably 1 to 300 nm, still more preferably 1 to 200 nm, and yet still more preferably 2 to 100 nm.

[0081] The average aspect ratio of the nanofibers (B) as a raw material prior to mixing with the base oil (A) is preferably

5 or more, more preferably 10 or more, still more preferably 15 or more, yet still more preferably 30 or more, even more preferably 50 or more, yet even more preferably 70 or more, further more preferably 90 or more, and yet further more preferably 100 or more.

[0082] In the description herein, the "thickness (d)" of the nanofibers (B) dispersed in the base oil (A) and the "thickness (d')" of the nanofibers (B) as a raw material prior to being blended in the base oil (A) as well as the aspect ratio of these nanofibers (B) each are a value as measured using an electron microscope or the like.

(Content of Nanofibers (B))

[0083] In the grease composition according to one embodiment of the present invention, as described above, the worked penetration is adjusted to an appropriate range even when the content of the nanofibers (B) is small. Specifically, in the grease composition according to one embodiment of the present invention, the content of the nanofibers (B) is preferably 0.1 to 20% by mass, more preferably 0.5 to 18% by mass, still more preferably 0.8 to 15% by mass, yet still more preferably 1.0 to 12% by mass, even more preferably 1.0 to 10% by mass, and yet even more preferably 1.0 to 9.0% by mass, based on the total amount (100% by mass) of the grease composition.

[0084] When the content of the nanofibers (B) is 0.1% by mass or more, it is easy to prepare a grease composition having a high dropping point.

[0085] On the other hand, when the content of the nanofibers (B) is 20% by mass or less, it is easy to prepare a grease composition excellent in wear resistance.

[0086] In addition, by adjusting the content of the nanofibers (B) within the aforementioned range, the worked penetration of the grease composition is also easily adjusted to an appropriate range.

[0087] Hereinafter, an embodiment in which the grease composition according to one embodiment of the present invention further contains an organic bentonite (C), which is a preferred embodiment, will be described.

<Organic Bentonite (C)>

[0088] The grease composition according to one embodiment of the present invention preferably further contains an organic bentonite (C) in addition to the base oil (A) and the nanofibers (B). The nanofibers (B) preferably contain cellulose nanofibers (B1).

[0089] As described above, when the grease composition contains the nanofibers (B), the nanofibers (B) are uniformly dispersed in the grease composition to form a higher-order structure. When the nanofibers (B) contain cellulose nanofibers (B1), the cellulose nanofibers (B1) are also uniformly dispersed in the grease composition to form a higher-order structure. In addition, the organic bentonite (C) is dispersed in the vicinity of the cellulose nanofibers (B1) that are uniformly dispersed because the hydrophilic surface (the surface having a hydrophilic group) adsorbs the hydrophilic group of the cellulose nanofibers (B1) or the hydrophilic surface thereof comes close to the hydrophilic group of the cellulose nanofibers (B1). As a result, the organic bentonite (C) is uniformly dispersed and arranged as if it surrounds the hydrophilic group of the cellulose nanofibers (B1).

[0090] Here, as described above, the nanofibers (B) have excellent mechanical stability. The cellulose nanofibers (B1) also have excellent mechanical stability. The organic bentonite (C) also has excellent mechanical stability. Therefore, the higher-order structure of the nanofibers (B) (cellulose nanofibers (B1)) and the organic bentonite (C) dispersed in the grease composition are stable against shearing. Therefore, the shear stability of the grease composition is improved, and the grease leakage prevention performance is improved.

[0091] In addition, since the cellulose nanofibers (B1) and the organic bentonite (C) are easily uniformly dispersed in the base oil (A), a grease composition having an appropriate worked penetration can be obtained even when the content of the cellulose nanofibers (B1) is small and the content of the organic bentonite (C) is also small, and thus the proportion of the base oil (A) in the grease composition can be increased. Thus, the lubricity of the grease composition is enhanced, and the energy transfer efficiency is also easily improved.

[0092] The organic bentonite (C) is uniformly dispersed and arranged to surround the hydrophilic group of the cellulose nanofibers (B1), which pseudo-hydrophobizes the cellulose nanofibers (B1) and provides excellent water resistance to the grease composition.

[0093] Here, "the content of the organic bentonite (C) is small" means that the content of the organic bentonite (C) is 0.01 to 15% by mass, preferably 0.1 to 10% by mass, and more preferably 1.0 to 8.0% by mass, based on the total amount (100% by mass) of the grease composition.

[0094] When the content of the organic bentonite is 0.01% by mass or more, it is easy to prepare a grease composition having more excellent water resistance.

[0095] On the other hand, when the content of the organic bentonite is 15% by mass or less, it is easy to prepare a grease composition excellent in energy transfer efficiency.

[0096] When the grease composition according to one embodiment of the present invention contains the organic

bentonite (C), the content of the cellulose nanofibers (B1) is preferably 60 to 100% by mass, more preferably 70 to 100% by mass, still more preferably 80 to 100% by mass, and yet still more preferably 90 to 100% by mass, based on the total amount of the nanofibers (B), from the viewpoint of maximizing the effect obtained by containing the organic bentonite (C).

[0097] In the grease composition according to one embodiment of the present invention, the total content of the base oil (A), the nanofibers (B), and the organic bentonite (C) is preferably 50% by mass or more, more preferably 60% by mass or more, still more preferably 70% by mass or more, yet still more preferably 80% by mass or more, and even more preferably 90% by mass or more, based on the total amount (100% by mass) of the grease composition.

[0098] In the grease composition according to one embodiment of the present invention, the content ratio [(B1)/(C)] of the cellulose nanofibers (B1) to the organic bentonite (C) is preferably 0.05 to 5.0, more preferably 0.1 to 2.0, and still more preferably 0.1 to 1.0, in terms of mass ratio, from the viewpoint of obtaining a grease composition that is more excellent in leakage prevention performance and energy transfer efficiency as well as excellent in water resistance.

[0099] The organic bentonite (C) is one prepared by modifying the crystal surface of a clay mineral, montmorillonite through treatment with a quaternary ammonium compound.

[0100] Not specifically limited, the quaternary ammonium compound may be any one capable of modifying the crystal surface of a clay mineral, montmorillonite, and examples thereof include dimethylalkylammonium such as dimethyloctadecylammonium; trimethylalkylammonium such as trimethyloctadecylammonium; and trialkylbenzylammonium. Among these, dimethylalkylammonium such as dimethyloctadecylammonium is preferred.

[0101] The quaternary ammonium compound may be used alone or in combination of two or more kinds thereof.

[0102] In addition, the organic bentonite (C) may be used alone or in combination of two or more kinds thereof.

[0103] In general, the organic bentonite (C) is cleaved when subjected to shear in the presence of a polar compound in a base oil and functions as a thickener. However, bentonite such as an organic bentonite is difficult to uniformly disperse in a base oil. Consequently, in general, a large amount of bentonite is blended in a grease composition using bentonite as a thickener (bentonite grease) to control the worked penetration of the composition. Specifically, bentonite is generally blended in an amount of 18% by mass or more, further 20% by mass or more, relative to the total amount (100% by mass) of the grease composition.

[0104] On the other hand, the grease composition according to one embodiment of the present invention enables the organic bentonite (C) to be uniformly dispersed in the base oil by using the cellulose nanofibers (B1) and the organic bentonite (C) in combination.

[0105] A method for producing an organic bentonite is disclosed in detail, for example, in JP 62-83108 A and JP 53-72792 A.

<Various Additives>

[0106] The grease composition according to one embodiment of the present invention may further contain various additives that are blended in general greases composition within a range where the effects of the present invention are not impaired.

[0107] Examples of the various additives include an extreme pressure agent, a rust inhibitor, an antioxidant, a detergent dispersant, a corrosion inhibitor, an anti-foaming agent, and a metal deactivator.

[0108] These various additives may be used either alone or in combination of two or more kinds thereof.

[0109] The grease composition according to one embodiment of the present invention may contain a dispersant and water used in grease formation within a range where the grease state may be maintained.

[0110] Examples of the dispersant include compounds exemplified in the method for producing the grease composition of the present invention described later.

[0111] In the grease composition according to one embodiment of the present invention, the total content of the dispersant and water is preferably 0 to 60% by mass, more preferably 0 to 30% by mass, still more preferably 0 to 10% by mass, and yet still more preferably 0 to 5% by mass, based on the total amount (100% by mass) of the grease.

(Extreme Pressure Agent)

[0112] Examples of the extreme pressure agent include one or more selected from a phosphorus extreme pressure agent and a sulfur-phosphorus extreme pressure agent.

[0113] Examples of the phosphorus extreme pressure agent include one or more phosphates selected from orthophosphate, hydrogen phosphate, polyphosphate, phosphite, and metaphosphate.

[0114] Examples of the polyphosphate include pyrophosphate (diphosphate), tripolyphosphate, and tetrapolyphosphate.

[0115] The phosphate is preferably an alkali metal salt. Preferable examples of the alkali metal salt include a sodium salt, a potassium salt and a lithium salt, and a sodium salt is particularly preferable.

[0116] Examples of the sulfur-phosphorus extreme pressure agent include one or more selected from a thiophosphoric

acid ester and an amine salt of a thiophosphoric acid ester.

[0117] Examples of the thiophosphoric acid ester include a monothiophosphoric acid ester, a dithiophosphoric acid ester, a trithiophosphoric acid ester, a monothiophosphorous acid ester, a dithiophosphorous acid ester, and a trithiophosphorous acid ester, and among these, a trithiophosphoric acid ester is preferable.

[0118] Examples of the trithiophosphoric acid ester include trialkyl phosphorothionates such as tributyl phosphorothionate, tripentyl phosphorothionate, trihexyl phosphorothionate, triheptyl phosphorothionate, trioctyl phosphorothionate, trinonyl phosphorothionate, tridecyl phosphorothionate, triundecyl phosphorothionate, tripentadecyl phosphorothionate, and trihexadecyl phosphorothionate; triaryl phosphorothionates such as triphenyl phosphorothionate, tricresyl phosphorothionate, and trixylenyl phosphorothionate; and tris(alkylphenyl)phosphorothionates such as tris(n-propylphenyl)phosphorothionate, tris(isopropylphenyl)phosphorothionate, tris(n-butylphenyl)phosphorothionate, tris(isobutylphenyl)phosphorothionate, tris(s-butylphenyl)phosphorothionate, tris(t-butylphenyl)phosphorothionate, and tris(2,4-C₆H₃(C₁₀H₇)₂O)thiophosphate.

[0119] Examples of the amine salt of the thiophosphoric acid ester include the amine salts of the thiophosphoric acid esters exemplified above.

[0120] The content of the extreme pressure agent contained in the grease composition according to one embodiment of the present invention is preferably 0.01 to 5.0% by mass, more preferably 0.1 to 3.0% by mass, and still more preferably 0.5 to 2.0% by mass, based on the total amount (100% by mass) of the grease composition.

[0121] Examples of the extreme pressure agent other than the aforementioned extreme pressure agent include organic molybdenum.

[0122] However, the grease composition according to one embodiment of the present invention preferably has a low content of molybdenum atoms. Specifically, the content of molybdenum atoms is preferably less than 50 ppm% by mass, more preferably less than 10 ppm% by mass, still more preferably less than 1 ppm% by mass, and yet still more preferably no molybdenum atoms, based on the total amount (100% by mass) of the grease composition.

(Rust Inhibitor)

[0123] Examples of the rust inhibitor include a carboxylic acid-based rust inhibitor, an amine-based rust inhibitor, and a carboxylate-based rust inhibitor.

[0124] When the grease composition according to one embodiment of the present invention contains a rust inhibitor, the content of the rust inhibitor is preferably 0.1 to 10.0% by mass, more preferably 0.3 to 8.0% by mass, and still more preferably 1.0 to 5.0% by mass, based on the total amount (100% by mass) of the grease composition.

(Antioxidant)

[0125] Examples of the antioxidant include an amine-based antioxidant, a phenol-based antioxidant, a sulfur-based antioxidant, and zinc dithiophosphate.

[0126] In the case where the grease composition according to one embodiment of the present invention contains an antioxidant, the content of the antioxidant is preferably 0.05 to 10% by mass, more preferably 0.1 to 7% by mass, and still more preferably 0.2 to 5% by mass, based on the total amount (100% by mass) of the grease composition.

(Detergent Dispersant, Corrosion Inhibitor, Anti-foaming Agent, Metal Deactivator)

[0127] Examples of the detergent dispersant include a succinimide, and a boron-based succinimide.

[0128] Examples of the corrosion inhibitor include a benzotriazole-based compound, and a thiazole-based compound.

[0129] Examples of the anti-foaming agent include a silicone-based compound, and a fluorinated silicone-based compound.

[0130] Examples of the metal deactivator include benzotriazole.

[0131] In the case where the grease composition according to one embodiment of the present invention contains these additives, the content of each of these additives is preferably 0.01 to 20% by mass, more preferably 0.1 to 10% by mass, and still more preferably 0.2 to 5% by mass, based on the total amount (100% by mass) of the grease composition.

[Characteristics of Grease Composition of the Present Invention]

(Worked Penetration)

[0132] The worked penetration at 25°C of the grease composition according to one embodiment of the present invention is preferably 220 to 440, more preferably 240 to 400, still more preferably 250 to 380, and yet still more preferably 270 to 360.

[0133] The grease composition according to one embodiment of the present invention is a grease composition that is excellent in the leakage prevention performance of the grease composition and is excellent in the energy transfer efficiency and the leakage prevention performance even when the worked penetration at 25°C is adjusted to the aforementioned range.

(Shear Stability)

[0134] With respect to the grease composition according to one embodiment of the present invention, the change in worked penetration in a roll stability test measured and calculated by the method described in Examples below is preferably 50 or less, more preferably 40 or less, still more preferably 30 or less, and yet still more preferably 20 or less.

(Torque Transmission Efficiency)

[0135] The grease composition according to one embodiment of the present invention has a torque transmission efficiency, which is an index of energy transfer efficiency, of preferably 60.0% or more, more preferably 63.0% or more, and still more preferably 66.0% or more, as measured and calculated by the method described in Examples below.

[Method for Producing Grease Composition of the Present Invention]

[0136] The method for producing the grease composition of the present invention is not particularly limited, but includes, for example, the following step (1), and the step (2) is performed as necessary.

Step (1): a step of preparing a liquid mixture in which nanofibers (B) having a thickness (d') of 1 to 500 nm are dispersed in a base oil (A).

Step (2): a step of removing an unnecessary component from the liquid mixture.

[0137] The nanofibers (B) are one or more types selected from cellulose nanofibers (B1) and modified cellulose nanofibers (B2).

[0138] In the grease composition obtained through such steps, the aggregation of the nanofibers (B) is suppressed, and the nanofibers having a thickness (d) of 1 to 500 nm can be dispersed in the base oil (A) while maintaining the fiber shape. As a result, a higher-order structure is formed by the nanofibers (B) in the base oil, and the nanofibers (B) can be uniformly dispersed in the base oil (A). Therefore, by adding a small amount of the nanofibers (B), a grease composition having an appropriate worked penetration can be prepared, and a grease composition excellent in both leakage prevention performance and energy transfer efficiency can be obtained.

[0139] Hereinunder the steps (1) to (2) are described.

<Step (1)>

[0140] The step (S1a) is a step of preparing a liquid mixture in which nanofibers (B) having a thickness (d') of 1 to 500 nm are dispersed in a base oil (A).

[0141] Details of the nanofibers (B) and the base oil (A) used in the step (S1a) are as described above.

[0142] The "thickness (d)" as referred to herein expresses the thickness of the nanofibers (B) as a raw material prior to being blended in the base oil (A) as described above, and a preferred range of the "thickness (d)" is the same as described above.

[0143] In one embodiment of the present invention, as the nanofibers (B), powdered cellulose nanofibers dispersible in water, an organic solvent, or the base oil (A) may be used, or a dispersion in which the nanofibers are dispersed in water, an organic solvent, or the base oil (A) may be used. Alternatively, nanofibers may be formed by applying shear in the base oil (A). When an aqueous dispersion in which the nanofibers (B) are dispersed in water or an organic solvent dispersion in which the nanofibers (B) are dispersed in an organic solvent is used, the solid content concentration of the dispersion having the nanofibers (B) blended therein is usually 0.1 to 70% by mass, preferably 0.1 to 65% by mass, more preferably 0.1 to 60% by mass, still more preferably 0.5 to 55% by mass, and yet still more preferably 1.0 to 50% by mass, based on the total amount (100% by mass) of the dispersion.

[0144] The dispersion can be prepared by blending the nanofibers (B) in water or an organic solvent, optionally blending a dispersant or the like when the aqueous dispersion is used, and sufficiently stirring the mixture manually or with a stirrer.

[0145] The dispersant is preferably one or more selected from aprotic polar solvents, such as N,N-dimethylformamide (DMF), N,N-dimethylacetamide (DMAc), and N-methylpyrrolidone (NMP); alcohols, such as propanol, ethylene glycol, propylene glycol, and hexylene glycol; and surfactants, such as a polyglycerin fatty acid ester, a sucrose fatty acid ester, a citric acid monoglyceride, a diacetyltartaric acid monoglyceride, a polyoxyethylene sorbitan acid ester, and sorbitan

acid ester.

[0146] When the aqueous dispersion is used, the blending amount of the dispersant in the liquid mixture that is prepared in the step (S1a) is preferably 0.1 to 50% by mass, more preferably 0.5 to 40% by mass, still more preferably 1.0 to 30% by mass, yet still more preferably 1.0 to 20% by mass, and even more preferably 1.0 to 10% by mass, based on the total amount (100% by mass) of the liquid mixture.

[0147] When the aqueous dispersion or the organic solvent dispersion is used, the blending amount of water or the organic dispersion in the liquid mixture that is prepared in the step (S1a) is preferably 1 to 60% by mass, more preferably 3 to 50% by mass, and still more preferably 5 to 40% by mass, based on the total amount (100% by mass) of the liquid mixture.

[0148] When the aqueous dispersion is used, the blending ratio of water to the dispersion medium (water/dispersion medium) in the liquid mixture that is prepared in the step (S1a) is preferably 0.01 to 600, more preferably 0.05 to 400, still more preferably 0.1 to 300, and yet still more preferably 0.2 to 200 in terms of mass ratio.

[0149] By sufficiently stirring the liquid mixture manually or with a stirrer, a liquid mixture in which the nanofibers (B) having a thickness (d') of 1 to 500 nm are dispersed in the base oil (A) can be prepared.

[0150] The liquid mixture in which the nanofibers (B) having a thickness (d') of 1 to 500 nm are dispersed in the base oil (A) can also be prepared by directly dispersing the nanofibers (B) in the base oil (A) or by applying shear to a nanofiber raw material in the base oil (A) to form nanofibers.

<Step (2)>

[0151] The step (2) is a step of removing an unnecessary component from the liquid mixture prepared in the step (1).

[0152] The unnecessary component is one or more selected from water, an organic solvent, and a dispersant in the liquid mixture.

[0153] However, these components may not be completely removed if the grease composition can maintain the state of grease.

[0154] As a method for removing one or more selected from water, an organic solvent, and a dispersant in the liquid mixture, a method of heating the liquid mixture and evaporating and removing the same is preferable.

[0155] As the conditions for the evaporation and removal, it is preferable to set the temperature range in consideration of the boiling point of one or more selected from an organic solvent and a dispersant in an environment at a pressure of 0.001 to 0.1 MPa, and to heat. The heating temperature is, for example, 0 to 100°C.

[0156] According to the step (2), a grease composition is prepared.

[0157] When a grease composition containing the organic bentonite (C) and further other additives is prepared, the organic bentonite (C) and further other additives may be mixed in the liquid mixture in the step (1), or may be mixed in the grease composition prepared in the step (2) and then subjected to a treatment such as homogenization using a roll mill or the like.

<Use of Grease Composition of the Present Invention>

[0158] The grease composition of the present invention is excellent in both leakage prevention performance and energy transfer efficiency.

[0159] Therefore, the grease composition according to one embodiment of the present invention can be suitably used for a speed reducer included in an industrial robot or the like and a speed increaser included in a wind power generation facility.

[0160] Examples of the speed reducer and the speed increaser include a speed reducer including a gear mechanism and a speed increaser including a gear mechanism. However, the object to which the grease composition according to one embodiment of the present invention is applied is not limited to a speed reducer including a gear mechanism and a speed increaser including a gear mechanism, and the grease composition can also be applied to, for example, a traction drive or the like.

[0161] Further, according to one embodiment of the present invention, there is provided a speed reducer or a speed increaser having the grease composition of the present invention at a lubricating portion.

[0162] Furthermore, according to one embodiment of the present invention, there is provided a lubricating method for lubricating a lubricating portion of a speed reducer or a speed increaser with the grease composition of the present invention.

[0163] Furthermore, the nanofibers (B) have a low environmental load and are excellent in safety for human bodies. Therefore, the grease composition of the present invention can be suitably used for a food machine including a speed reducer, a food machine including a speed increaser, and the like.

[0164] In addition, the organic bentonite (C) also has a low environmental load and is excellent in safety for human bodies. Therefore, the grease composition according to one embodiment of the present invention containing the organic

bentonite (C) can also be suitably used for a food machine including a speed reducer, a food machine including a speed increaser, and the like.

[0165] Accordingly, in one embodiment of the present invention, there is provided a food machine including a speed reducer or a speed increaser having the grease composition of the present invention at a lubricating portion.

[0166] Further, in one embodiment of the present invention, there is provided a lubrication method including lubricating a lubricating portion of a food machine including a speed reducer or a speed increaser with the grease composition of the present invention.

Examples

[0167] The present invention is described in more detail by reference to Examples given below, but it should be construed that the present invention is by no means limited to these Examples.

[Property Values of Raw Materials]

[0168] Property values of raw materials were determined according to the following methods.

(1) Thickness and Aspect Ratio of Nanofibers (B)

[0169] Ten arbitrarily selected hydrophilic nanofibers were each measured with respect to the thickness and the length thereof by using a transmission electron microscope (TEM), and a value as calculated from "length/thickness" was defined as an "aspect ratio" of the hydrophilic nanofibers measured.

(2) 40°C Kinematic Viscosity, Viscosity Index

[0170] The measurement and calculation were performed in conformity with JIS K2283:2000.

[Examples 1 to 5 and Comparative Examples 1 to 3]

[0171] In Examples 1 to 5 and Comparative Examples 1 to 3, the following raw materials were used.

<Base oil (A)>

[0172] A low-viscosity base oil (A1) and a high-viscosity base oil (A2) were combined to prepare a mixed base oil having a 40°C kinematic viscosity of 60 mm²/s and a viscosity index of 135.

- Low-viscosity base oil (A1): poly- α -olefin, 40°C kinematic viscosity 46 mm²/s, viscosity index 137
- High-viscosity base oil (A2): poly- α -olefin, 40°C kinematic viscosity 403 mm²/s, viscosity index 150

<Thickener>

[0173]

- Nanofibers (B): product name "BiNFi-s", manufactured by Sugino Machine Limited (aqueous dispersion containing cellulose nanofibers (CNF) having a degree of polymerization of 600 (thickness (d') = 20 to 50 nm (average value: 35 nm), aspect ratio = 100 or more (average value: 100 or more)). Hereinafter, the dispersion is also referred to as "dispersion of cellulose nanofibers (B1)".
- Organic bentonite (C): product name "BARAGEL (registered trademark) 3000", manufactured by Elementis Specialties, Inc.
- Lithium stearate
- Aluminum stearate

<Extreme pressure agent>

[0174]

- Phosphorus extreme pressure agent: sodium polyphosphate
- Sulfur-phosphorus extreme pressure agent: triphenyl phosphorothionate

<Dispersant>

[0175]

- 5 • Sorbitan acid ester

<Antioxidant>

[0176]

- 10 • Amine-based antioxidant

<Example 1>

15 **[0177]** 75 parts by mass (in this, CNF amount: 7.5 parts by mass) of the cellulose nanofiber (B1) dispersion, 89.7 parts by mass of the base oil (A), and 0.8 parts by mass of the dispersant were mixed, and sufficiently stirred at 25°C to prepare a liquid mixture. Then, the liquid mixture was heated up to 150°C to evaporate and remove water from the liquid mixture.

20 **[0178]** Next, after cooling to room temperature (25°C), 1.0 part by mass of the phosphorus extreme pressure agent, 0.5 parts by mass of the sulfur-phosphorus extreme pressure agent, and 0.5 parts by mass of the antioxidant were added to the liquid mixture and sufficiently stirred. Thereafter, a homogenization treatment was performed using a three-roll mill to prepare a grease composition having a formulation shown in Example 1 of Table 1.

[0179] In Example 1, (B1)/(C) = 7.5/0 (mass ratio).

25 <Example 2>

[0180] 40 parts by mass (in this, CNF amount: 4.0 parts by mass) of the cellulose nanofiber (B1) dispersion, 84.8 parts by mass of the base oil (A), and 0.4 parts by mass of the dispersant were mixed, and sufficiently stirred at 25°C to prepare a liquid mixture. Then, the liquid mixture was heated up to 150°C to evaporate and remove water from the liquid mixture.

30 **[0181]** Next, after cooling to room temperature (25°C), 8.8 parts by mass of the organic bentonite (C), 1.0 part by mass of the phosphorus extreme pressure agent, 0.5 parts by mass of the sulfur-phosphorus extreme pressure agent, and 0.5 parts by mass of the antioxidant were added to the liquid mixture and sufficiently stirred. Thereafter, a homogenization treatment was performed using a three-roll mill to prepare a grease composition having a formulation shown in Example 2 of Table 1.

35 **[0182]** In Example 2, (B1)/(C) = 0.45 (mass ratio).

<Example 3>

40 **[0183]** A grease composition having a formulation shown in Example 3 of Table 1 was prepared under the same conditions as in Example 2 except that the phosphorus extreme pressure agent was not added and the blending amount of the base oil (A) was changed to 85.8 parts by mass.

[0184] In Example 3, (B1)/(C) = 0.45 (mass ratio).

45 <Example 4>

[0185] 28 parts by mass (in this, CNF amount: 2.8 parts by mass) of the cellulose nanofiber (B1) dispersion, 88.4 parts by mass of the base oil (A), and 0.3 parts by mass of the dispersant were mixed, and sufficiently stirred at 25°C to prepare a liquid mixture. Then, the liquid mixture was heated up to 150°C to evaporate and remove water from the liquid mixture.

50 **[0186]** Next, after cooling to room temperature (25°C), 6.5 parts by mass of the organic bentonite (C), 1.0 part by mass of the phosphorus extreme pressure agent, 0.5 parts by mass of the sulfur-phosphorus extreme pressure agent, and 0.5 parts by mass of the antioxidant were added to the liquid mixture and sufficiently stirred. Thereafter, a homogenization treatment was performed using a three-roll mill to prepare a grease composition having a formulation shown in Example 4 of Table 1.

55 **[0187]** In Example 4, (B1)/(C) = 0.43 (mass ratio).

<Example 5>

[0188] A grease composition having a formulation shown in Example 5 of Table 1 was prepared under the same conditions as in Example 3 except that the sulfur-phosphorus extreme pressure agent was not added and the blending amount of the base oil (A) was changed to 86.3 parts by mass.

[0189] In Example 5, (B1)/(C) = 0.45 (mass ratio).

<Comparative Examples 1 to 3>

[0190] The components shown in Table 1 were mixed in the proportions shown in Table 1 to prepare lubricant compositions of Comparative Examples 1 to 3.

[Evaluation]

[0191] The grease compositions thus prepared were evaluated as follows.

<Evaluation of worked penetration>

[0192] Measurement was carried out at 25°C in conformity with JIS K2220 7:2013.

<Evaluation of shear stability>

[0193] The change in penetration in the roll stability test was measured by the method described in ASTM D 1831. However, the temperature and time were changed to 80°C and 20 hours, respectively.

[0194] The smaller the change in penetration in the roll stability test, the more excellent the shear stability and the more excellent the grease leakage prevention performance.

<Evaluation of energy transfer efficiency>

[0195] Fig. 1 is a schematic view of an apparatus used for measuring torque transmission efficiency as an index of energy transfer efficiency in this embodiment.

[0196] The measuring apparatus 100 shown in Fig. 1 is formed by connecting an input side motor unit 111, an input side torque measuring device 112, an input side speed reducer 113 (manufactured by Harmonic Drive Systems Inc., product name "CSG-40-100-2UH"), an output side torque measuring device 122, an output side speed reducer 123 (manufactured by Nabtesco Corporation, product name "RV-125V"), and an output side motor unit 121 in this order.

[0197] A grease-filled case (case internal temperature: 30°C) included in the input side speed reducer 113 of the measuring apparatus 1 shown in Fig. 1 was filled with 140 g of a mixed grease, the measuring apparatus 100 was operated under conditions of a load torque of 240 Nm and an input side rotation speed of 1600 rpm, the rotation speeds and torques on the input side and the output side were measured, the torque transmission efficiency was calculated from the following equation (1), and the energy transfer efficiency was evaluated.

$$(\text{torque transmission efficiency (\%)}) = (\text{output side torque (Nm)}) / [(\text{input side torque (Nm)}) \times (\text{reduction ratio})] \times 100 \quad (1)$$

[0198] The reduction ratio is 100.

[0199] The torque transmission efficiency is an index indicating an amount of loss until input energy is output. A lower torque transmission efficiency means a larger energy loss, and a higher torque transmission efficiency means a smaller energy loss.

[0200] The results are shown in Table 1.

Table 1

	Unit	Example 1	Example 2	Example 3	Example 4	Example 5	Comparative Example 1	Comparative Example 2	Comparative Example 3
Base oil (A)	% by mass	89.7	84.8	85.8	88.4	86.3	80.5	89.0	87.7
Thickener	Cellulose nanofibers (B)	7.5	4.0	4.0	2.8	4.0	-	-	-
	Organic bentonite (C)	-	8.8	8.8	6.5	8.8	18.0	-	-
	Lithium stearate	-	-	-	-	-	-	9.5	-
	Aluminum stearate	-	-	-	-	-	-	-	10.8
	Thickener total amount	7.5	12.8	12.8	9.3	12.8	18.0	9.5	10.8
Extreme pressure agent	Phosphorus extreme pressure agent	1.0	1.0	-	1.0	-	1.0	1.0	1.0
	Sulfur-phosphorus extreme pressure agent	0.5	0.5	0.5	0.5	-	-	-	-
Dispersant	Sorbitan acid ester	0.8	0.4	0.4	0.3	0.4	-	-	-
Antioxidant	Amine-based antioxidant	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Evaluation	Worked penetration (JIS K2220 7:2013)	272	294	281	350	277	268	283	309
	Shear stability (change in worked penetration in the roll stability test)	-6	9	7	18	9	110	60	130
	Energy transfer efficiency	70.1	69.5	68.9	70.5	66.4	53.4	59.8	54.6

[0201] From Table 1, the following can be seen.

[0202] It can be seen that the grease compositions of Examples 1 to 5 have an appropriate worked penetration and are excellent in shear stability and energy transfer efficiency. Therefore, it can be seen that the grease compositions are excellent in leakage prevention performance and energy transfer efficiency.

[0203] On the other hand, it can be seen that the grease composition in which the organic bentonite is blended without blending the nanofibers (B) as in the grease composition of Comparative Example 1 has an appropriate worked penetration, but is inferior in shear stability and energy transfer efficiency.

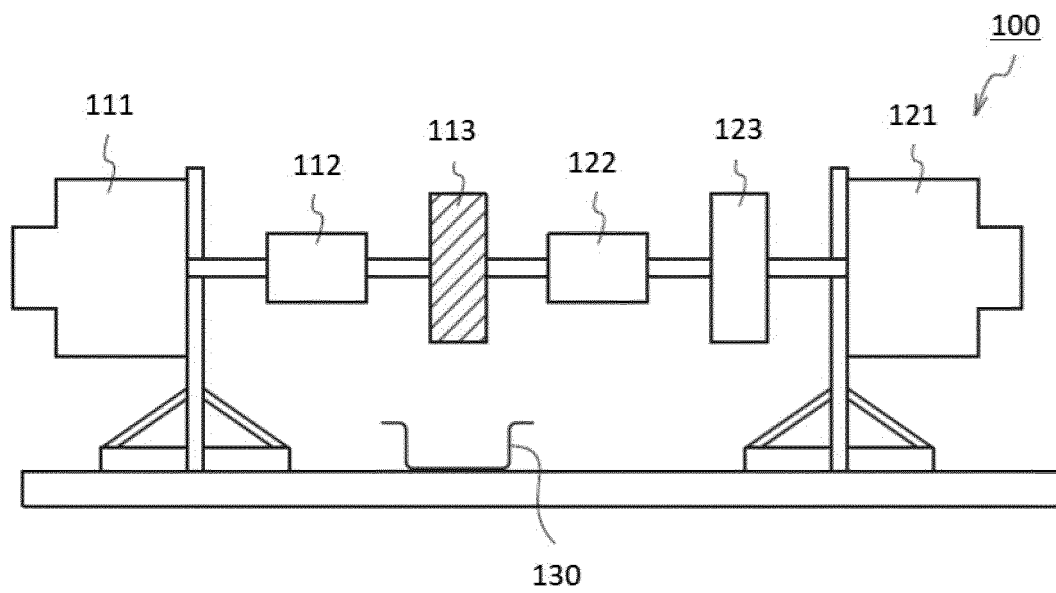
[0204] Also, it can be seen that the grease compositions using lithium stearate or aluminum stearate as a thickener as in the grease compositions of Comparative Examples 2 and 3 have an appropriate worked penetration, but are inferior in shear stability and energy transfer efficiency.

[0205] As a result of confirming whether or not the thickness of the cellulose nanofibers (B1) changed before and after the preparation of the grease composition in Example 1, it was confirmed that the thickness hardly changed before and after the preparation. From this, it is considered that there is little difference between the "thickness (d) of the nanofibers (B)" dispersed in the base oil and the "thickness (d') of the nanofibers (B)" as a raw material prior to being blended in the base oil, and the two are substantially the same.

Claims

1. A grease composition used for a speed reducer and a speed increaser, comprising: a base oil (A); and nanofibers (B) having a thickness (d) of 1 to 500 nm, wherein the nanofibers (B) are one or more selected from cellulose nanofibers (B1) and modified cellulose nanofibers (B2).
2. The grease composition according to claim 1, wherein the content of the nanofibers (B) is 0.1 to 20% by mass based on the total amount of the grease composition.
3. The grease composition according to claim 1 or 2, further comprising an organic bentonite (C), wherein the nanofibers (B) contain the cellulose nanofibers (B1).
4. The grease composition according to claim 3, wherein the content of the organic bentonite (C) is 0.01 to 15% by mass based on the total amount of the grease composition.
5. The grease composition according to claim 3 or 4, wherein a content ratio [B1/C] of the cellulose nanofibers (B1) to the organic bentonite (C) is 0.05 to 5.0 in terms of mass ratio.
6. The grease composition according to any one of claims 1 to 5, wherein the base oil (A) is a mixed base oil containing a low-viscosity base oil (A1) having a kinematic viscosity at 40°C of 5 to 150 mm²/s and a high-viscosity base oil (A2) having a kinematic viscosity at 40°C of 200 to 1000 mm²/s.
7. The grease composition according to any one of claims 1 to 6, wherein the grease composition has a worked penetration at 25°C of 220 to 440.
8. The grease composition according to any one of claims 1 to 7, wherein the grease composition is used in a food machine including a speed reducer or a food machine including a speed increaser.
9. A lubrication method, comprising lubricating a lubricating portion of a speed reducer or a speed increaser with the grease composition according to any one of claims 1 to 7.
10. A lubrication method, comprising lubricating a lubricating portion of a food machine including a speed reducer or a speed increaser with the grease composition according to any one of claims 1 to 7.

[Fig. 1]



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2020/007973

A. CLASSIFICATION OF SUBJECT MATTER

C10M 169/06(2006.01)i; C10N 20/02(2006.01)n; C10N 30/00(2006.01)n; C10N 40/06(2006.01)n; C10N 50/10(2006.01)n; C10M 113/16(2006.01)n; C10M 119/20(2006.01)i

FI: C10M119/20; C10M169/06; C10M113/16; C10N30:00 Z; C10N20:02; C10N50:10; C10N40:06

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)

C10M169/06; C10N20/02; C10N30/00; C10N40/06; C10N50/10; C10M113/16; C10M119/20

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	1971-2020
Registered utility model specifications of Japan	1996-2020
Published registered utility model applications of Japan	1994-2020

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2017-180526 A (SEIKO EPSON CORP.) 05.10.2017 (2017-10-05) claims, paragraphs [0006], [0031], [0053], [0056]-[0061], [0063]	1-10
Y	claims, paragraphs [0006], [0031], [0053], [0056]-[0061], [0063]	1-2, 6-10
Y	WO 2016/175258 A1 (IDEMITSU KOSAN CO., LTD.) 03.11.2016 (2016-11-03) claims, paragraphs [0015], [0070], examples	1-2, 6-10
Y	JP 2017-082024 A (IDEMITSU KOSAN CO., LTD.) 18.05.2017 (2017-05-18) claims, paragraphs [0017], [0060], examples	1-2, 6-10
A	JP 2017-210612 A (NTN CORPORATION) 30.11.2017 (2017-11-30) claims	1-10



Further documents are listed in the continuation of Box C.



See patent family annex.

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

Date of the actual completion of the international search
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Name and mailing address of the ISA/
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3-4-3, Kasumigaseki, Chiyoda-ku,
Tokyo 100-8915, Japan

Authorized officer

Telephone No.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2020/007973

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2018-193524 A (IDEMITSU KOSAN CO., LTD.) 06.12.2018 (2018-12-06) claims, paragraph [0002]	1-10
P, A	WO 2019/189239 A1 (IDEMITSU KOSAN CO., LTD.) 03.10.2019 (2019-10-03) claims, paragraph [0075]	1-10

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application no.
PCT/JP2020/007973

Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
JP 2017-180526 A	05 Oct. 2017	(Family: none)	
WO 2016/175258 A1	03 Nov. 2016	US 2018/0079983 A1 claims, paragraphs [0035], [0143], examples EP 3290497 A1 CN 107532104 A	
JP 2017-082024 A	18 May 2017	(Family: none)	
JP 2017-210612 A	30 Nov. 2017	WO 2017/200098 A1 claims	
JP 2018-193524 A	06 Dec. 2018	WO 2018/216569 A1 claims, paragraph [0002]	
WO 2019/189239 A1	03 Oct. 2019	(Family: none)	

REFERENCES CITED IN THE DESCRIPTION

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