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(54) **COMPOSITION, LUBRICATING OIL COMPOSITION AND GREASE COMPOSITION**

(57) A composition containing metal based nanoparticles (X), a polymer (Y), and a dispersion medium, the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group, the metal based nanoparticles (X) and the polymer (Y) being dispersed in the dispersion medium, the composition being used as an additive composition for a lubricating oil composition, an additive composition for a grease composition, a lubricating oil composition, or a grease composition.

The polymer (Y) is one or more kind selected from the group consisting of a polymer (Y1) that contains two or more kinds selected from the group consisting of a constitutional unit derived from the following monomer (ya), a constitutional unit derived from the following monomer

(yb), and a constitutional unit derived from the following monomer (yc), and a polymer (Y2) that contains a constitutional unit derived from the following monomer (yb) and does not contain a constitutional unit derived from the following monomer (ya) and a constitutional unit derived from the following monomer (yc): monomer (ya): an olefin having 12 to 30 carbon atoms, monomer (yb): a vinyl monomer having a nitrogen-containing heterocyclic group, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms, and monomer (yc): a vinyl monomer having a nitrogen-containing heterocyclic group, all hydrogen atoms of which are not substituted by a chain-like alkyl group having 12 to 30 carbon atoms.

Description

Technical Field

5 **[0001]** The present invention relates to a composition, a lubricating oil composition, and a grease composition.

Background Art

10 **[0002]** Metal based particles that are dispersed in a lubricant base oil have a potential of imparting various functions to the lubricant base oil. Therefore, a lubricating oil composition containing a lubricant base oil having metal based particles blended therein has been variously investigated.

15 **[0003]** In recent years, a lubricating oil composition containing a lubricant base oil having metal based nanoparticles, which are extremely fine metal based particles, blended therein is being also investigated. For example, PTLs 1 and 2 describe that the addition of a combination of metal based nanoparticles and an acrylate based polymer to a lubricant base oil can significantly favorably disperse the metal based nanoparticles in the lubricant base oil, and a lubricating oil composition having improved extreme pressure capability and friction reduction capability imparted thereto can be provided.

Citation List

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Patent Literatures

[0004]

25 PTL 1: JP 2021-512188 A
PTL 2: JP 2021-512189 A

Summary of Invention

30 **[0005]** However, the techniques described in PTLs 1 and 2 are insufficient in the dispersibility of the metal based nanoparticles under a high temperature environment (for example, 200°C or more), which is expected in the use of a lubricating oil composition. Furthermore, the techniques described in PTLs 1 and 2 are also insufficient in the dispersibility of the metal based nanoparticles in grease.

35 **[0006]** Under the circumstances, a problem to be solved by the present invention is to provide a composition that is improved in dispersibility of metal based nanoparticles, a lubricating oil composition, and a grease composition.

Solution to Problem

40 **[0007]** The present invention provides the following items [1] to [4].

[1] A composition containing metal based nanoparticles (X), a polymer (Y), and a dispersion medium,

the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,

45 the metal based nanoparticles (X) and the polymer (Y) being dispersed in the dispersion medium, the composition being used as an additive composition for a lubricating oil composition, an additive composition for a grease composition, a lubricating oil composition, or a grease composition.

50 [2] A method for producing a composition, including the following step (1),

step (1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and a dispersion medium, the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group.

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[3] A lubricating oil composition containing metal based nanoparticles (X), a polymer (Y), and a lubricant base oil,

the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-

containing heterocyclic group,
the metal based nanoparticles (X) and the polymer (Y) being dispersed in the lubricant base oil.

[4] A grease composition containing metal based nanoparticles (X), a polymer (Y), and grease,

the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,
the metal based nanoparticles (X) and the polymer (Y) being dispersed in the grease.

Advantageous Effects of Invention

[0008] The present invention can provide a composition that is improved in dispersibility of metal based nanoparticles, a lubricating oil composition, and a grease composition.

Description of Embodiments

[0009] In the description herein, the upper limit values and the lower limit values of the numerical ranges can be optionally combined. For example, in the case where numerical ranges of "A to B" and "C to D" are described, numerical ranges of "A to D" and "C to B" are also encompassed in the scope of the present invention.

[0010] In the description herein, the numerical range of "lower limit value to upper limit value" means the lower limit value or more and the upper limit value or less, unless otherwise indicated.

[0011] In the description herein, the numerical values described in the examples are numerical values that can be used as the upper limit value or the lower limit value.

[Embodiments of Composition]

[0012] The composition of the present embodiment contains metal based nanoparticles (X), a polymer (Y), and a dispersion medium.

[0013] The polymer (Y) is a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group.

[0014] The composition of the present embodiment contains the metal based nanoparticles (X) and the polymer (Y) that are dispersed in the dispersion medium, and is used as an additive composition for a lubricating oil composition, an additive composition for a grease composition, a lubricating oil composition, or a grease composition.

[0015] The present inventors have made earnest investigations for solving the problem. As a result, it has been found that the composition having the aforementioned configuration is significantly excellent in dispersibility of metal based nanoparticles.

[0016] In more detail, it has been found that the composition having the aforementioned configuration is excellent in dispersibility of metal based nanoparticles even under a high temperature environment (for example, 200°C or more), which is expected in the use of a lubricating oil composition, and is also excellent in dispersibility of metal based nanoparticles in a grease composition.

[0017] While details of the mechanism by which the effects of the present invention are exerted is not clear, it is estimated that the structure of the polymer (Y) used in the present embodiment contributes to the improvement of the dispersibility of the metal based nanoparticles.

[0018] The "metal based nanoparticles (X)", the "polymer (Y)", and the "dispersion medium" constituting the composition of the present embodiment will be described in detail below.

<Metal based Nanoparticles (X)>

[0019] The composition of the present embodiment contains metal based nanoparticles (X).

[0020] In the present embodiment, the metal based nanoparticles (X) are particles having an average particle diameter in nm (nanometer) order, i.e., less than 1 μm.

[0021] The average particle diameter of the metal based nanoparticles (X) is preferably 800 nm or less, more preferably 600 nm or less, further preferably 500 nm or less, still further preferably 400 nm or less, still more further preferably 300 nm or less, and even further preferably 200 nm or less, from the standpoint of the enhancement of the dispersibility and the like. The average particle diameter of the metal based nanoparticles (X) is generally 10 nm or more.

[0022] The "average particle diameter of the metal based nanoparticles (X)" in the present embodiment means an average particle diameter in a dispersion state in a dispersion medium at 25°C that is obtained by performing the measurement method in the section "1. Investigation on Dispersibility of Metal based Nanoparticles" in the examples

described later.

[0023] In the description herein, the "average particle diameter" means an integrated value, Z-average particle diameter, in a particle size distribution obtained by the dynamic light scattering method.

[0024] In the present embodiment, the "metal based" is a concept that encompasses not only metals, but also metal oxides, metal nitrides, metal sulfides, metal carbides, metal borides, and the like.

[0025] However, boron is excluded from the metal elements constituting the metal borides.

[0026] In the present embodiment, the metal based nanoparticles (X) preferably contains one or more kind of metal based nanoparticles (X1) selected from the group consisting of metal nanoparticles containing one or more kind of a metal element (x1) selected from the group consisting of transition metal elements, and metal elements and semimetal elements of Groups 12 to 15, nanoparticles containing an oxide of the metal element (x1), nanoparticles containing a nitride of the metal element (x1), nanoparticles containing a sulfide of the metal element (x1), nanoparticles containing a carbide of the metal element (x1), and nanoparticles containing a boride of the metal element (x1), from the standpoint of the enhancement of the dispersibility and the like.

[0027] The content of the metal based nanoparticles (X1) in the metal based nanoparticles (X) is preferably 50% by mass to 100% by mass, more preferably 60% by mass to 100% by mass, further preferably 70% by mass to 100% by mass, still further preferably 80% by mass to 100% by mass, and still more further preferably 90% by mass to 100% by mass, based on the total amount of the metal based nanoparticles (X), from the standpoint of the enhancement of the dispersibility and the like.

[0028] Specific examples of the metal element (x1) will be described below.

[0029] Examples of the transition metal element include first transition metal elements, such as scandium (Sc), titanium (Ti), vanadium (V), chromium (Cr), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), and copper (Cu); second transition metal elements, such as yttrium (Y), zirconium (Zr), niobium (Nb), molybdenum (Mo), technetium (Tc), ruthenium (Ru), rhodium (Rh), palladium (Pd), and silver (Ag); and third transition metal elements, such as lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd), promethium (Pm), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), thulium (Tm), ytterbium (Yb), lutetium (Lu), hafnium (Hf), tantalum (Ta), tungsten (W), rhenium (Re), osmium (Os), iridium (Ir), platinum (Pt), and gold (Au).

[0030] Examples of the metal elements of Groups 12 to 15 include metal elements of Group 12, such as zinc (Zn), cadmium (Cd), and mercury (Hg); metal elements of Group 13, such as aluminum (Al), gallium (Ga), indium (In), and thallium (Tl); metal elements of Group 14, such as germanium (Ge), tin (Sn), and lead (Pb); and metal elements of Group 15, such as bismuth (Bi).

[0031] Examples of the semimetal elements of Groups 12 to 15 include boron (B), silicon (Si), germanium (Ge), arsenic (As), antimony (Sb), and tellurium (Te).

[0032] In the metal element (x1), one or more kind selected from the group consisting of titanium (Ti), manganese (Mn), iron (Fe), nickel (Ni), copper (Cu), zirconium (Zr), molybdenum (Mo), tungsten (W), zinc (Zn), boron (B), aluminum (Al), and the like is preferred from the standpoint of the availability, the economic efficiency, and the like.

[0033] The metal based nanoparticles (X1) are preferably one or more kind selected from the group consisting of titania (TiO₂), manganese sulfide (MnS), iron oxide (FeO and Fe₂O₃), nickel oxide (NiO), copper oxide (CuO and Cu₂O), zirconia (ZrO₂), molybdenum disulfide (MoS₂), tungsten disulfide (WS₂), zinc oxide (ZnO), boron nitride (BN), titanium nitride (TiN), alumina (Al₂O₃), and the like from the standpoint of the availability, the economic efficiency, and the like.

[0034] Among these, one or more kind selected from the group consisting of zirconia (ZrO₂), tungsten disulfide (WS₂), zinc oxide (ZnO), and titanium nitride (TiN) is preferred from the standpoint of the enhancement of the wear resistance of the composition (e.g., the lubricating oil composition and the grease composition).

<Polymer (Y)>

[0035] In the present embodiment, the polymer (Y) is a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group.

[0036] In the case where the polymer (Y) does not have a chain-like alkyl group having 10 to 28 carbon atoms, the oil solubility (i.e., the solubility in a low polarity oil, such as a mineral oil and a hydrocarbon oil) cannot be secured.

[0037] It is estimated that the nitrogen-containing heterocyclic group assumes the function exhibiting the dispersibility of the metal based nanoparticles (X) in the polymer (Y). Therefore, in the case where the polymer (Y) does not have a nitrogen-containing heterocyclic group, the function exhibiting excellent dispersibility of the metal based nanoparticles (X) cannot be secured.

[0038] The number of carbon atoms of the chain-like alkyl group is preferably 11 to 26, more preferably 12 to 24, and further preferably 13 to 22, from the standpoint of further facilitating the enhancement of the oil solubility of the polymer (Y).

[0039] Specific examples of the chain-like alkyl group include a decyl group, an undecyl group, a dodecyl group, a tridecyl group, a tetradecyl group, a pentadecyl group, a hexadecyl group, a heptadecyl group, an octadecyl group, a nonadecyl group, an icosyl group, a heneicosyl group, a docosyl group, a tricosyl group, a tetracosyl group, a pentacosyl

group, a hexacosyl group, a heptacosyl group, an octacosyl group, a nonacosyl group, and an eicosyl group.

[0040] The chain-like alkyl group may have a linear chain structure or a branched chain structure, and preferably has a linear chain structure from the standpoint of further facilitating the enhancement of the oil solubility of the polymer (Y).

[0041] Examples of the nitrogen-containing heterocyclic group include a 5-membered ring, a 6-membered ring, a bicyclic compound, and a multimer each containing a nitrogen atom. Specific examples of the nitrogen-containing heterocyclic group include monovalent groups obtained by removing one hydrogen atom from nitrogen-containing heterocyclic rings, such as a pyrrole ring, a pyrroline ring, a pyrrolidine ring, a pyrrolidone ring, a pyrazole ring, a pyrazoline ring, a pyrazolidine ring, an imidazole ring, an imidazoline ring, an imidazolidine ring, a triazole ring, a tetrazole ring, an oxazole ring, an oxazoline ring, an oxazolidine ring, an isoxazole ring, an isoxazoline ring, an isoxazolidine ring, a thiazole ring, a thiazoline ring, a thiazolidine ring, an isothiazole ring, an isothiazoline ring, an isothiazolidine ring, a pyridine ring, a piperidine ring, a pyridazine ring, a pyrimidine ring, a pyrazine ring, a piperazine ring, a triazine ring, a morpholine ring, a thiomorpholine ring, an ϵ -caprolactam ring, an indole ring, an indazole ring, a benzimidazole ring, a quinoline ring, an isoquinoline ring, a purine ring, a bipyridine ring, and a terpyridine ring.

[0042] The nitrogen-containing heterocyclic group is preferably a monovalent group obtained by removing one hydrogen atom from a pyrrolidone ring from the standpoint of facilitating the enhancement of the effects of the present invention.

[0043] The polymer (Y) is preferably one or more kind selected from the group consisting of a polymer (Y1) that contains two or more kinds selected from the group consisting of a constitutional unit derived from the following monomer (ya), a constitutional unit derived from the following monomer (yb), and a constitutional unit derived from the following monomer (yc), and a polymer (Y2) that contains a constitutional unit derived from the following monomer (yb) and does not contain a constitutional unit derived from the following monomer (ya) and a constitutional unit derived from the following monomer (yc), from the standpoint of facilitating the enhancement of the effects of the present invention.

Monomer (ya): an olefin having 12 to 30 carbon atoms

Monomer (yb): a vinyl monomer having a nitrogen-containing heterocyclic group, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms

Monomer (yc): a vinyl monomer having a nitrogen-containing heterocyclic group, all hydrogen atoms of which are not substituted by a chain-like alkyl group having 12 to 30 carbon atoms

[0044] The polymer (Y1) and the polymer (Y2) will be described in detail below.

<Polymer (Y1)>

[0045] The polymer (Y1) contains two or more kinds selected from the group consisting of a constitutional unit derived from the following monomer (ya), a constitutional unit derived from the following monomer (yb), and a constitutional unit derived from the following monomer (yc).

Monomer (ya): an olefin having 12 to 30 carbon atoms

Monomer (yb): a vinyl monomer having a nitrogen-containing heterocyclic group, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms

Monomer (yc): a vinyl monomer having a nitrogen-containing heterocyclic group, all hydrogen atoms of which are not substituted by a chain-like alkyl group having 12 to 30 carbon atoms

[0046] In the present embodiment, the polymer (Y1) may contain a constitutional unit other than the constitutional unit derived from the monomer (ya), the constitutional unit derived from the monomer (yb), and the constitutional unit derived from the monomer (yc), in such a range that does not impair the effects of the present invention.

[0047] In the present embodiment, the total content of two or more kinds of constitutional units selected from the group consisting of the constitutional unit derived from the monomer (ya), the constitutional unit derived from the monomer (yb), and the constitutional unit derived from the monomer (yc) in the polymer (Y1) is preferably 50% by mol to 100% by mol, more preferably 60% by mol to 100% by mol, further preferably 70% by mol to 100% by mol, still further preferably 80% by mol to 100% by mol, and still more further preferably 90% by mol to 100% by mol, based on the total constitutional units of the polymer (Y1), from the standpoint of the securement of the oil solubility required for the polymer (Y1) and the dispersibility of the metal nanoparticles (X).

[0048] The monomer (ya), the monomer (yb), and the monomer (yc) will be described in detail below.

(Monomer (ya), Constitutional Unit (YA))

[0049] The monomer (ya) used in the present embodiment is an olefin having 12 to 30 carbon atoms.

[0050] A constitutional unit (YA) derived from the monomer (ya) mainly assumes the function exhibiting the oil solubility (i.e., the solubility in a low polarity oil, such as a mineral oil and a hydrocarbon oil) in the polymer (Y1).

[0051] The number of carbon atoms of the monomer (ya) is preferably 13 to 28, more preferably 14 to 26, further preferably 15 to 24, and still further preferably 16 to 22, from the standpoint of facilitating the enhancement of the oil solubility of the polymer (Y1).

[0052] Specific examples of the preferred compound as the monomer (ya) include dodecene, tridecene, tetradecene, pentadecene, hexadecene, heptadecene, octadecene, nonadecene, icosene (eicosene), heneicosene (heneicosene), docosene, tricosene, tetracosene, pentacosene, hexacosene, heptacosene, octacosene, nonacosene, and triacontene.

[0053] The monomer (ya) may have a linear chain structure or a branched chain structure, and preferably has a linear chain structure from the standpoint of further facilitating the enhancement of the oil solubility of the polymer (Y1).

[0054] The double bond site of the monomer (ya) is preferably the 1-position to 3-position, more preferably the 1-position or the 2-position, and further preferably the 1-position, from the standpoint of further facilitating the enhancement of the oil solubility of the polymer (Y1). In other words, the monomer (ya) is preferably an α -olefin.

[0055] Therefore, the monomer (ya) preferably contains a linear α -olefin, and more preferably contains a linear α -olefin having 12 to 30 carbon atoms. The preferred number of carbon atoms of the linear α -olefin is as described above.

[0056] One kind of the monomer (ya) may be used alone, or two or more kinds thereof may be used in combination. Therefore, the polymer (Y1) may contain one kind of the constitutional unit (YA) derived from the monomer (ya) alone, or may contain two or more kinds thereof.

(Monomer (yb), Constitutional Unit (YB))

[0057] The monomer (yb) used in the present embodiment is a vinyl monomer having a nitrogen-containing heterocyclic group, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms. The chain-like alkyl group having 12 to 30 carbon atoms in a constitutional unit (YB) derived from the monomer (yb) mainly assumes the function exhibiting the oil solubility (i.e., the solubility in a low polarity oil, such as a mineral oil and a hydrocarbon oil) in the polymer (Y1).

[0058] It is estimated that the nitrogen-containing heterocyclic group in the constitutional unit (YB) derived from the monomer (yb) assumes the function exhibiting the dispersibility of the metal based nanoparticles (X) in the polymer (Y1), and it is also estimated that the nitrogen-containing heterocyclic group assumes the function exhibiting the stability of the polymer (Y1) under a high temperature environment.

[0059] The number of carbon atoms of the chain-like alkyl group that substitutes at least one hydrogen atom of the nitrogen-containing heterocyclic group is preferably 13 to 28, more preferably 14 to 26, further preferably 15 to 24, and still further preferably 16 to 22, from the standpoint further facilitating the enhancement of the oil solubility of the polymer (Y2).

[0060] Examples of the preferred chain-like alkyl group include a dodecyl group, a tridecyl group, a tetradecyl group, a pentadecyl group, a hexadecyl group, a heptadecyl group, an octadecyl group, a nonadecyl group, an icosyl group (an eicosyl group), a heneicosyl group (a heneicosyl group), a docosyl group, a tricosyl group, a tetracosyl group, a pentacosyl group, a hexacosyl group, a heptacosyl group, an octacosyl group, a nonacosyl group, and a triacontyl group.

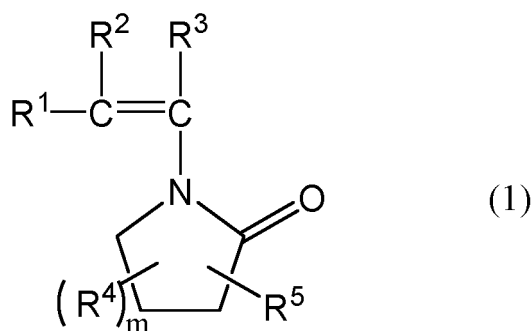
[0061] The chain-like alkyl group may have a linear chain structure or a branched chain structure, and preferably has a linear chain structure from the standpoint of further facilitating the enhancement of the oil solubility of the polymer (Y1).

[0062] Examples of the nitrogen-containing heterocyclic group include the same groups as described for the nitrogen-containing heterocyclic group that the polymer (Y) has.

[0063] The nitrogen-containing heterocyclic group is preferably a monovalent group obtained by removing one hydrogen atom from a pyrrolidone ring from the standpoint of facilitating the enhancement of the effects of the present invention.

[0064] Therefore, the monomer (yb) is preferably a vinyl monomer having a monovalent group obtained by removing one hydrogen atom from a pyrrolidone ring, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms.

[0065] Specifically, the monomer (yb) is preferably a compound represented by the following general formula (1).



[0066] In the general formula (1), R^1 , R^2 , and R^3 each independently represents a hydrogen atom or a chain-like alkyl group having 1 to 2 carbon atoms, R^4 represents a chain-like alkyl group having 1 to 5 carbon atoms, R^5 represents a chain-like alkyl group having 12 to 30 carbon atoms, and m represents an integer of 0 to 2.

[0067] R^1 , R^2 , and R^3 preferably represent hydrogen atoms from the standpoint of further facilitating the enhancement of the effects of the present invention. It is preferred that $m = 0$ from the same standpoint. The preferred embodiment of the chain-like alkyl group having 12 to 30 carbon atoms that can be selected as R^5 are as described above.

[0068] One kind of the monomer (yb) may be used alone, or two or more kinds thereof may be used in combination. Therefore, the polymer (Y1) may contain one kind of the constitutional unit (YB) derived from the monomer (yb) alone, or may contain two or more kinds thereof.

(Monomer (yc), Constitutional Unit (YC))

[0069] The monomer (yc) used in the present embodiment is a vinyl monomer having a nitrogen-containing heterocyclic group, all hydrogen atoms of which are not substituted by a chain-like alkyl group having 12 to 30 carbon atoms.

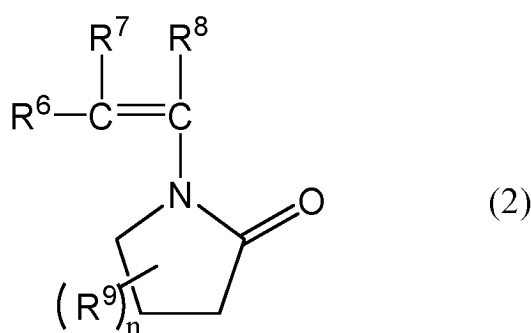
[0070] It is estimated that a constitutional unit (YC) derived from the monomer (yc) assumes the function exhibiting the excellent dispersibility of the metal based nanoparticles (X) in the polymer (Y1).

[0071] Examples of the nitrogen-containing heterocyclic group include the same groups as described for the nitrogen-containing heterocyclic group that the polymer (Y) has.

[0072] The nitrogen-containing heterocyclic group is preferably a monovalent group obtained by removing one hydrogen atom from a pyrrolidone ring from the standpoint of facilitating the enhancement of the effects of the present invention.

[0073] Therefore, the monomer (yc) is preferably a vinyl monomer having a monovalent group obtained by removing one hydrogen atom from a pyrrolidone ring, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms.

[0074] Specifically, the monomer (yc) is preferably a compound represented by the following general formula (2).



[0075] In the general formula (2), R^6 , R^7 , and R^8 each independently represents a hydrogen atom or a chain-like alkyl group having 1 to 2 carbon atoms, R^9 represents a chain-like alkyl group having 1 to 5 carbon atoms, and n represents an integer of 0 to 3.

[0076] R^6 , R^7 , and R^8 preferably represent hydrogen atoms from the standpoint of further facilitating the enhancement of the effects of the present invention. It is preferred that $n = 0$ from the same standpoint.

[0077] One kind of the monomer (yc) may be used alone, or two or more kinds thereof may be used in combination. Therefore, the polymer (Y1) may contain one kind of the constitutional unit (YC) derived from the monomer (yc) alone, or may contain two or more kinds thereof.

(Preferred Embodiments of Polymer (Y1))

[0078] Examples of the polymer (Y1) include polymers containing constitutional units derived from the following monomers.

- monomer (ya) and monomer (yb)
- monomer (ya) and monomer (yc)
- monomer (yb) and monomer (yc)
- monomer (ya), monomer (yb), and monomer (yc)

[0079] With the combinations above, the polymer (Y1) can be a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group.

[0080] Among the polymers, the polymer (Y1) is preferably a polymer containing the constitutional unit (YA) derived from the monomer (ya) and the constitutional unit (YC) derived from the monomer (yc) from the standpoint of the balance between the securement of the oil solubility and the securement of the dispersibility of the metal based nanoparticles (X).

(Content Ratio of Constitutional Unit (YA) and Constitutional Unit (YC))

[0081] In the case where the polymer (Y1) is a polymer that contains the constitutional unit (YA) derived from the monomer (ya) and the constitutional unit (YC) derived from the monomer (yc), the content ratio ((YA)/(YC)) of the constitutional unit (YA) derived from the monomer (ya) and the constitutional unit (YC) derived from the monomer (yc) in terms of molar ratio is preferably 10/90 to 95/5, more preferably 40/60 to 90/10, and further preferably 60/40 to 90/10, from the standpoint of facilitating the securement of the balance between the oil solubility of the polymer (Y1) and the dispersibility of the metal based nanoparticles (X).

<Polymer (Y2)>

[0082] The polymer (Y2) contains a constitutional unit derived from the monomer (yb) and does not contain a constitutional unit derived from the monomer (ya) and a constitutional unit derived from the monomer (yc).

[0083] The polymer (Y2) containing the constitutional unit (YB) derived from the monomer (yb) is a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group, and is excellent in balance between the securement of the oil solubility and the securement of the dispersibility of the metal based nanoparticles (X).

[0084] In the present embodiment, the polymer (Y2) may be constituted only by the constitutional unit (YB) derived from the monomer (yb), and may contain a constitutional unit other than the constitutional unit (YB) in such a range that does not impair the effects of the present invention. However, the polymer (Y2) does not contain a constitutional unit derived from the monomer (ya) and a constitutional unit derived from the monomer (yc), and is clearly distinguished from the polymer (Y1) in this point.

[0085] In the present embodiment, the content of the constitutional unit (YB) in the polymer (Y2) is preferably 70% by mol to 100% by mol, more preferably 80% by mol to 100% by mol, and further preferably 90% by mol to 100% by mol, based on the entire constitutional units of the polymer (Y2), from the standpoint of the securement of the oil solubility required for the polymer (Y2) and the dispersibility of the metal nanoparticles (X).

[0086] The preferred embodiments of the monomer (yb) have been described above, and the description thereof is omitted therein.

<Synthesis Method of Polymer (Y)>

[0087] The polymer (Y) can be appropriately synthesized, for example, in such a synthesis method that the monomer components in the prescribed proportions are subjected to homopolymerization or copolymerization.

[0088] The polymer (Y) used may be a commercially available product. Examples of the commercially available product include Antaron V-220 (available from Ashland Japan, Ltd.) and Antaron V-216 (available from Ashland Japan, Ltd.).

<Content Ratio ((Y)/(X)) of Polymer (Y) and Metal based Nanoparticles (X)>

[0089] In the composition of the present embodiment, the content ratio ((Y)/(X)) of the polymer (Y) and the metal based nanoparticles (X) in terms of mass ratio is preferably 0.2 or more, and more preferably 0.3 or more, from the standpoint of facilitating the favorable dispersion of the metal based nanoparticles (X) in the dispersion medium.

[0090] The content ratio ((Y)/(X)) of the polymer (Y) and the metal based nanoparticles (X) in terms of mass ratio is

preferably 10 or less, more preferably 6 or less, and further preferably 3 or less, from the standpoint of facilitating the favorable dispersion of the metal based nanoparticles (X) in the dispersion medium, while preventing the excessive use of the polymer (Y).

<Dispersion Medium>

[0091] Examples of the dispersion medium include one or more kind of a liquid dispersion medium selected from the group consisting of an organic solvent and a lubricant base oil, and grease.

[0092] Examples of the organic solvent include an apolar organic solvent, such as an alkane. The use of an apolar organic solvent facilitates the mixing and dispersion of the composition in a lubricant base oil in the case where the composition is blended in a low polar lubricant base oil.

[0093] Examples of the lubricant base oil include low polar or apolar lubricant base oils, for example, a mineral oil; a hydrocarbon based synthetic oil, such as a polyolefin, an isoparaffin, an alkylbenzene, and an alkylnaphthalene; a GTL base oil obtained through isomerization of wax (gas-to-liquid (GTL) wax) produced from natural gas by the Fischer-Tropsch process or the like; and an ether oil, such as an alkylated diphenyl ether and a polyphenyl ether.

[0094] Examples of the grease include soap based grease, such as calcium soap grease, calcium complex grease, sodium soap grease, aluminum soap grease, aluminum complex grease, lithium soap grease, and lithium complex grease; a non-soap based inorganic grease, such as organized bentonite grease and silica gel grease; and a non-soap based organic grease, such as polyurea grease.

<Content of Metal based Nanoparticles (X) in Composition>

[0095] The content of the metal based nanoparticles (X) in the composition is not particularly limited, as far as securing the favorable dispersibility of the metal based nanoparticles (X) in the dispersion medium, and is preferably 0.005% by mass or more, more preferably 0.010% by mass or more, further preferably 0.050% by mass or more, still further preferably 0.10% by mass or more, and still more further preferably 0.50% by mass or more, and is preferably 30% by mass or less, more preferably 20% by mass or less, further preferably 10% by mass or less, and still further preferably 5% by mass or less, based on the total amount of the composition.

[0096] The upper limit values and the lower limit values of the numerical ranges can be optionally combined. Specifically, the content thereof is preferably 0.005% by mass to 30% by mass, more preferably 0.010% by mass to 20% by mass, further preferably 0.050% by mass to 10% by mass, still further preferably 0.10% by mass to 5% by mass, and still more further preferably 0.50% by mass to 5% by mass.

[Method of producing Composition]

[0097] The method for producing the composition of the present embodiment is not particularly limited, and examples thereof include a production method including the following step (1).

[0098] Step (1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and one or more kind of a liquid dispersion medium selected from the group consisting of an organic solvent and a lubricant base oil

[0099] The "precursor of metal based nanoparticles (X)" means a raw material (in the state before dispersing in the dispersion medium) of the metal based nanoparticles (X) constituting the composition of the present embodiment. The "precursor of metal based nanoparticles (X)" is an aggregate of many primary particles aggregated, and the aggregate can be converted to the state where the metal based nanoparticles (X) having the prescribed particle diameter are dispersed in the dispersion medium, through pulverization, trituration, cracking, or the like by performing the dispersing treatment.

[0100] Examples of the measure for performing the dispersing treatment in the step (1) include a kneading device, such as a roll mill and a kneader, an ultrasonic disperser, a high pressure homogenizer, such as a microfluidizer, a paint shaker, and a medium type disperser, such as a bead mill.

[0101] Among these, a medium type disperser is preferably used from the standpoint of facilitating the reduction of the particle diameter of the precursor of the metal based nanoparticles (X) (i.e., the standpoint of facilitating getting the particle diameter thereof close to the particle diameter of the primary particles).

[0102] In the case where the medium type disperser is used, the material of the medium is preferably ceramics, such as zirconia and titania, a polymer material, such as polyethylene and polyamide, a metal, and the like, in which zirconia is particularly preferred. The diameter of the medium is preferably 3 μm or more, more preferably 10 μm or more, and 20 μm or more, and is preferably 500 μm or less, more preferably 300 μm or less, and further preferably 100 μm or less, from the standpoint of making the precursor of the metal based nanoparticles (X) sufficiently fine.

[0103] The dispersing treatment time cannot be determined unconditionally since the appropriate dispersing treatment time may vary depending on the size of the medium type disperser, the amount of the medium charged, and the amount of

the raw material charged. As one example, in the case where approximately 150 g of the medium is charged in a vessel having a capacity of 100 mL, and the dispersing treatment is performed with 20 to 40 g of the raw material charged therein, the rough target of the dispersing treatment time is preferably 0.3 hour or more, and more preferably 1 hour or more, from the standpoint of making the precursor of the metal based nanoparticles (X) sufficiently fine, and is preferably 10 hours or less, and more preferably 5 hours or less, from the standpoint of the production efficiency of the fine particle aqueous dispersion liquid.

[0104] The shearing force in the dispersing treatment cannot be determined unconditionally since the appropriate shearing force in the dispersing treatment may vary depending on the size of the medium type disperser, the amount of the medium charged, and the amount of the raw material charged. As one example, in the case where approximately 150 g of the medium is charged in a vessel having a capacity of 100 mL, and the dispersing treatment is performed with 20 to 40 g of the raw material charged therein, the dispersing treatment is performed by agitating under condition of preferably 1,000 rpm or more, more preferably 1,300 rpm or more, and further preferably 1,500 rpm or more, and of preferably 5,000 rpm or less, more preferably 4,000 rpm or less, and further preferably 3,000 rpm or less.

[Application of Composition]

[0105] The composition of the present embodiment is excellent in dispersibility of the metal based nanoparticles (X) under high temperature environment (for example, 200°C or more), which is expected in the use of a lubricating oil composition. Therefore, the composition is useful as an additive composition for a lubricating oil composition.

[0106] The composition of the present embodiment is excellent in dispersibility of the metal based nanoparticles (X) in grease. Therefore, the composition is useful as an additive composition for a grease composition.

[0107] Accordingly, the present embodiment provides a use method including using the composition of the present embodiment as an additive composition for a lubricating oil composition or a grease composition.

[0108] In the case where the composition of the present embodiment is used as an additive composition for a lubricating oil composition or an additive composition for a grease composition, the dispersion medium is preferably one or more kind selected from the group consisting of an organic solvent and a lubricant base oil.

[0109] In the case where the composition of the present embodiment is used as an additive composition for a lubricating oil composition or an additive composition for a grease composition, the additive composition can impart excellent wear resistance to a lubricating oil composition or a grease composition. Therefore, the composition of the present embodiment is useful as a wear resistant agent.

[0110] Accordingly, the present embodiment provides a use method including using the composition of the present embodiment as a wear resistant agent.

[0111] The composition of the present embodiment is also useful as a lubricating oil composition or a grease composition as described below.

[Lubricating Oil Composition]

[0112] The composition of the present embodiment can also be used as a lubricating oil composition.

[0113] Accordingly, the present embodiment provides the following lubricating oil composition:

a lubricating oil composition containing metal based nanoparticles (X), a polymer (Y), and a lubricant base oil, the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group, the metal based nanoparticles (X) and the polymer (Y) being dispersed in the lubricant base oil.

[0114] In the lubricating oil composition of the present embodiment, the content of the metal based nanoparticles (X) is preferably 0.005% by mass or more, more preferably 0.010% by mass or more, further preferably 0.050% by mass or more, still further preferably 0.10% by mass or more, still more further preferably 0.50% by mass or more, and even further preferably 0.80% by mass or more, based on the total amount of the lubricating oil composition, from the standpoint of enhancing the dispersibility of the metal based nanoparticles (X) in the lubricant base oil and the standpoint of enhancing the wear resistance. The content thereof is preferably 5% by mass or less, more preferably 3% by mass or less, and further preferably 2% by mass or less.

[0115] The upper limit values and the lower limit values of the numerical ranges can be optionally combined. Specifically, the content thereof is preferably 0.005% by mass to 5% by mass, more preferably 0.010% by mass to 5% by mass, further preferably 0.050% by mass to 3% by mass, still further preferably 0.10% by mass to 3% by mass, still more further preferably 0.50% by mass to 2% by mass, and even further preferably 0.80% by mass to 2% by mass.

[0116] In the lubricating oil composition of the present embodiment, the content of the polymer (Y) is preferably 0.01% by mass or more, more preferably 0.05% by mass or more, further preferably 0.10% by mass or more, still further preferably

0.20% by mass or more, and still more further preferably 0.30% by mass or more, based on the total amount of the lubricating oil composition, from the standpoint of enhancing the dispersibility of the metal based nanoparticles (X) in the lubricant base oil. The content thereof is preferably 10% by mass or less, more preferably 5% by mass or less, and further preferably 3% by mass or less.

[0117] The upper limit values and the lower limit values of the numerical ranges can be optionally combined. Specifically, the content thereof is preferably 0.01% by mass to 10% by mass, more preferably 0.05% by mass to 10% by mass, further preferably 0.10% by mass to 5% by mass, still further preferably 0.20% by mass to 5% by mass, and still more further preferably 0.30% by mass to 3% by mass.

[0118] In the lubricating oil composition of the present embodiment, the preferred embodiments of the metal based nanoparticles (X) and the polymer (Y), the preferred content ratio of the metal based nanoparticles (X) and the polymer (Y), and the like are as described for the composition of the present embodiment.

[0119] In the lubricating oil composition of the present embodiment, the average particle diameter of the metal based nanoparticles (X) is preferably less than 600 nm, more preferably 500 nm or less, further preferably 400 nm or less, still further preferably 300 nm or less, still more further preferably 200 nm or less, and even further preferably 150 nm or less, from the standpoint of facilitating the enhancement of the wear resistance, and is generally 10 nm or more.

<Lubricant Base Oil>

[0120] The lubricant base oil used may be appropriately the base oils exemplified as the dispersion medium in the composition of the present embodiment. However, the lubricant base oil may be a mixed base oil containing an additional synthetic oil or the like other than the base oils exemplified as the dispersion medium.

[0121] Examples of the additional synthetic oil include one or more kind selected from an ester, such as a polyol ester and a dibasic acid ester, and a polyalkylene glycol.

[0122] The kinematic viscosity at 100°C of the lubricant base oil is preferably in a range of 1.0 mm²/s to 50 mm²/s, more preferably in a range of 2.0 mm²/s to 30 mm²/s, and further preferably in a range of 3.0 mm²/s to 20 mm²/s. The viscosity index of the lubricant base oil is preferably 80 or more, more preferably 90 or more, and further preferably 100 or more.

[0123] The kinematic viscosity and the viscosity index of the lubricant base oil are values that are measured or calculated according to JIS K2283:2000.

<Additives>

[0124] The lubricating oil composition of the present embodiment may further contain the ordinary additives that have been blended in lubricating oil compositions, in such a range that does not deviate from the substance of the present invention. Examples of the additives include an antioxidant, an oiliness agent, a detergent dispersant, a viscosity index improver, a rust inhibitor, a metal deactivator, and an anti-foaming agent. One kind of the additives may be used alone, or two or more kinds thereof may be used in combination.

<Properties of Lubricating Oil Composition>

(Kinematic Viscosity and Viscosity Index)

[0125] The 100°C kinematic viscosity of the lubricating oil composition of the present embodiment is preferably 1.0 mm²/s to 50 mm²/s, more preferably 2.0 mm²/s to 30 mm²/s, and further preferably 3.0 mm²/s to 20 mm²/s.

[0126] The viscosity index of the lubricating oil composition of the present embodiment is preferably 90 or more, more preferably 100 or more, and further preferably 110 or more.

[0127] The kinematic viscosity and the viscosity index of the lubricating oil composition are values that are measured or calculated according to JIS K2283:2000.

(Wear Resistance)

[0128] The lubricating oil composition of the present embodiment preferably has a wear track diameter (average) in the wearing test (using the unheated lubricating oil composition) shown in the examples described later of preferably 470 μm or less, more preferably 460 μm or less, further preferably 450 μm or less, still further preferably 440 μm or less, and still more further preferably 430 μm or less.

[Application of Lubricating Oil Composition]

[0129] The lubricating oil composition of the present embodiment is excellent in wear resistance.

[0130] Therefore, the lubricating oil composition of the present embodiment can be favorably applied, for example, to a drive train fluid, for example, a gear fluid (such as a manual transmission fluid and a differential fluid), an automatic transmission fluid, a continuously variable transmission fluid (such as a belt CVT fluid and a toroidal CVT fluid), a power steering fluid, a shock absorber fluid, and an electric motor fluid; an internal combustion engine oil, such as a gasoline engine oil, a diesel engine oil, and a gas engine oil; a hydraulic fluid; a turbine oil; a compressor oil; a hydrodynamic bearing fluid; a roller bearing fluid; and a refrigerator oil, and can be preferably used by charging in devices used in these applications, as a lubricating oil composition that lubricates the components of the devices.

[Lubricating Method using Lubricating Oil Composition]

[0131] Preferred examples of the lubricating method using the lubricating oil composition of the present embodiment include a method for charging the lubricating oil composition in the device used in the application described above, and lubricating the components of the device.

[0132] The present embodiment also provides use including charging the lubricating oil composition in the device used in the application described above, and lubricating the components of the device.

[Method of producing Lubricating Oil Composition]

[0133] Examples of the method for producing the lubricating oil composition of the present embodiment include the following production methods 1 to 3.

(Production Method 1)

[0134] A method for producing a lubricating oil composition, including the following step (S1).

Step (S1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and a lubricant base oil

[0135] The ordinary additives that have been blended in lubricating oil compositions may be blended simultaneously with the step (S1) or after the step (S1).

(Production Method 2)

[0136] A method for producing a lubricating oil composition, including the following step (S2-1) and the following step (S2-2).

Step (S2-1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and an organic solvent, so as to prepare an additive composition for a lubricating oil composition

Step (S2-2): a step of mixing a lubricant base oil and the additive composition for a lubricating oil composition, and then vaporizing the organic solvent

[0137] The ordinary additives that have been blended in lubricating oil compositions may be blended simultaneously with the step (S2-2) or after the step (S2-2).

(Production Method 3)

[0138] A method for producing a lubricating oil composition, including the following step (S3-1) and the following step (S3-2).

Step (S3-1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and a lubricant base oil, so as to prepare an additive composition for a lubricating oil composition

Step (S3-2): a step of mixing a lubricant base oil and the additive composition for a lubricating oil composition

[0139] The ordinary additives that have been blended in lubricating oil compositions may be blended simultaneously with the step (S3-2) or after the step (S3-2).

[Grease Composition]

[0140] The composition of the present embodiment can also be used as a grease composition.

[0141] Accordingly, the present embodiment provides the following grease composition:

a grease composition containing metal based nanoparticles (X), a polymer (Y), and grease, the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group, the metal based nanoparticles (X) and the polymer (Y) being dispersed in the grease.

[0142] In the grease composition of the present embodiment, the content of the metal based nanoparticles (X) is preferably 1% by mass or more, more preferably 2% by mass or more, further preferably 3% by mass or more, based on the total amount of the grease composition, from the standpoint of enhancing the dispersibility of the metal based nanoparticles (X) in the grease and the standpoint of enhancing the wear resistance. The content thereof is preferably 10% by mass or less, more preferably 8% by mass or less, and further preferably 6% by mass or less.

[0143] The upper limit values and the lower limit values of the numerical ranges can be optionally combined. Specifically, the content thereof is preferably 1% by mass to 10% by mass, more preferably 2% by mass to 8% by mass, and further preferably 3% by mass to 6% by mass.

[0144] In the grease composition of the present embodiment, the content of the polymer (Y) is preferably 1% by mass or more, more preferably 2% by mass or more, and further preferably 3% by mass or more, based on the total amount of the grease composition, from the standpoint of enhancing the dispersibility of the metal based nanoparticles (X) in the grease. The content thereof is preferably 20% by mass or less, more preferably 16% by mass or less, and further preferably 12% by mass or less.

[0145] The upper limit values and the lower limit values of the numerical ranges can be optionally combined. Specifically, the content thereof is preferably 1% by mass to 20% by mass, more preferably 2% by mass to 16% by mass, and further preferably 3% by mass to 12% by mass.

[0146] In the grease composition of the present embodiment, the preferred embodiments of the metal based nanoparticles (X) and the polymer (Y), the preferred content ratio of the metal based nanoparticles (X) and the polymer (Y), and the like are as described for the composition of the present embodiment.

[0147] In the grease composition of the present embodiment, the metal based nanoparticles (X) are dispersed in the grease composition significantly homogeneously without uneven distribution. This can be elucidated from the effect of reducing the electric resistivity in dispersing titanium nitride nanoparticles or tungsten disulfide nanoparticles in the examples. It is estimated that in the grease composition of the present embodiment, the minute metal based nanoparticles (X) are dispersed significantly homogeneously, and as a result, a certain mutual interaction occurs among the proximate metal based nanoparticles (X) to form a high-level network of the minute metal based nanoparticles (X). It is considered that the high-level dispersion state cannot be achieved with the ordinary solid lubricant or the like. It is estimated that the high-level dispersion state favorably exhibits the effects of the metal based nanoparticles (X), such as the enhancement of the wear resistance and the reduction of the electric resistivity.

(Grease)

[0148] The grease used may be appropriately those exemplified as the dispersion medium in the composition of the present embodiment.

[0149] Among these, polyurea grease is preferably used from the standpoint of the heat resistance and the like.

[0150] In the polyurea grease, aliphatic diurea grease is more preferably used.

[0151] The lubricant base oil constituting the grease may be appropriately those exemplified as the dispersion medium in the present embodiment.

<Additives>

[0152] The grease composition of the present embodiment may contain the ordinary additives that have been blended in grease, in such a range that does not deviate from the substance of the present invention.

[0153] Examples of the additives include an antioxidant, a rust inhibitor, an extreme pressure agent, a thickener, a solid lubricant, a detergent dispersant, a corrosion inhibitor, and a metal deactivator.

[0154] One kind of the additives may be used alone, or two or more kinds thereof may be used in combination.

<Properties of Grease Composition>

(Wear Resistance)

[0155] The grease composition of the present embodiment preferably has a wear track diameter (average) in the wearing test shown in the examples described later of preferably 260 μm or less, more preferably 250 μm or less, and further preferably 240 μm or less.

(Friction Coefficient)

[0156] The grease composition of the present embodiment preferably has a friction coefficient in the wearing test shown in the examples described later of preferably 0.100 or less, more preferably 0.090 or less, further preferably 0.080 or less, and still further preferably 0.070 or less.

(Volume Resistivity)

[0157] The grease composition of the present embodiment preferably has a volume resistivity measured by the method shown in the examples described later of preferably less than $1.0 \times 10^{13} \Omega\text{cm}$, more preferably $1.5 \times 10^{12} \Omega\text{cm}$ or less, and further preferably $1.2 \times 10^{12} \Omega\text{cm}$ or less.

[Application of Grease Composition]

[0158] The grease composition of the present embodiment is excellent in wear resistance.

[0159] Therefore, the grease composition of the present embodiment can be favorably applied, for example, to bearings, such as a plain bearing, a roller bearing, an oil-retaining bearing, and a hydrodynamic bearing, a speed reducer, a gear, an internal combustion engine, a brake, a torque transmission device component, a hydraulic coupling, a compressor component, a chain, a hydraulic device component, a vacuum pump device component, a clock component, a hard disk component, a refrigerator component, a cutting machine component, a rolling machine component, drawing machine component, a rolling tool component, an automobile component, a forging machine component, a heat treatment device component, a thermal medium component, a cleaning machine component, a shock absorber component, and a sealing device component.

[0160] The grease composition of the present embodiment that contains the metal based nanoparticles (X) excellent in electroconductivity has a low electric resistivity and is excellent in electroconductivity. Therefore, the grease composition of the present embodiment can be used for preventing electrolytic corrosion. In other words, the grease composition of the present embodiment can be used as electrolytic corrosion preventing grease.

[0161] While representative examples of the metal based nanoparticles (X) excellent in electroconductivity include titanium nitride nanoparticles and tungsten disulfide nanoparticles, the metal based nanoparticles (X) excellent in electroconductivity are not limited thereto, and those constituted by a material excellent in electroconductivity can be appropriately used.

[Lubricating Method using Grease Composition]

[0162] Preferred examples of the lubricating method using the grease composition of the present embodiment include a method for charging the grease composition in the device used in the application described above, and lubricating the components of the device.

[0163] The present embodiment also provides use including charging the grease composition in the device used in the application described above, and lubricating the components of the device.

[Method of producing Grease Composition]

[0164] Examples of the method for producing the grease composition of the present embodiment include the following production methods 4 to 8. Among these, the following production methods 5 to 8 are preferred from the standpoint of the easiness in producing the grease composition and the like.

(Production Method 4)

[0165] A method for producing a grease composition, including the following steps (S4-1) and (S4-2).

Step (S4-1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and an organic solvent, so as to prepare an additive composition for a grease composition
 Step (S4-2): a step of mixing grease and the additive composition for a grease composition, and then vaporizing the organic solvent

[0166] The ordinary additives that have been blended in grease compositions may be blended simultaneously with the step (S4-2) or after the step (S4-2).

[0167] The grease and the additive composition for a grease composition are mixed, for example, with a roll mill.

(Production Method 5)

[0168] A method for producing a grease composition, including the following steps (S5-1) and (S5-2).

Step (S5-1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and a lubricant base oil, so as to prepare an additive composition for a grease composition
 Step (S5-2): a step of mixing grease and the additive composition for a grease composition

[0169] The ordinary additives that have been blended in grease compositions may be blended simultaneously with the step (S5-2) or after the step (S5-2).

[0170] The grease and the additive composition for a grease composition are mixed, for example, with a roll mill.

(Production Method 6)

[0171] A method for producing a grease composition, including the following step (S6-1), the following step (S6-2), and the following step (S6-3).

Step (S6-1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and an organic solvent, so as to prepare an additive composition 1 containing the organic solvent as a dispersion medium

Step (S6-2): a step of mixing a lubricant base oil and the additive composition 1, and then vaporizing the organic solvent, so as to prepare an additive composition 2 containing the lubricant base oil as a dispersion medium

Step (S6-3): a step of mixing grease and the additive composition 2

[0172] The ordinary additives that have been blended in grease compositions may be blended simultaneously with the step (S6-3) or after the step (S6-3).

[0173] The grease and the additive composition 2 are mixed, for example, with a roll mill.

(Production Method 7)

[0174] A method for producing a grease composition, including the following steps (S7-1) and (S7-2).

Step (S7-1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and a lubricant base oil, so as to prepare an additive composition containing the lubricant base oil as a dispersion medium

Step (S7-2): a step of producing grease from a lubricant base oil (α) and a thickening agent (β), in which the additive composition is used as the lubricant base oil (α), or the additive composition is mixed in the lubricant base oil (α)

[0175] The ordinary additives that have been blended in grease compositions may be blended simultaneously with the step (S7-2) or after the step (S7-2).

(Production Method 8)

[0176] A method for producing a grease composition, including the following step (S8-1), the following step (S8-2), and the following step (S8-3).

Step (S8-1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and an organic solvent, so as to prepare an additive composition 1 containing the organic solvent as a dispersion medium

Step (S8-2): a step of mixing a lubricant base oil and the additive composition 1, and then vaporizing the organic solvent, so as to prepare an additive composition 2 containing the lubricant base oil as a dispersion medium

Step (S8-3): a step of producing grease from a lubricant base oil (α) and a thickening agent (β), in which the additive composition 2 is used as the lubricant base oil (α), or the additive composition 2 is mixed in the lubricant base oil (α)

[0177] The ordinary additives that have been blended in grease compositions may be blended simultaneously with the step (S8-3) or after the step (S8-3).

[One Embodiment of Present Invention]

[0178] One embodiment of the present invention provides the following items [1] to [15].

[1] A composition containing metal based nanoparticles (X), a polymer (Y), and a dispersion medium,

the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,
the metal based nanoparticles (X) and the polymer (Y) being dispersed in the dispersion medium,
the composition being used as an additive composition for a lubricating oil composition, an additive composition for a grease composition, a lubricating oil composition, or a grease composition.

[2] The composition according to the item [1], in which the metal based nanoparticles (X) contains one or more kind of metal based nanoparticles (X1) selected from the group consisting of metal nanoparticles containing one or more kind of a metal element (x1) selected from the group consisting of transition metal elements, and metal elements and semimetal elements of Groups 12 to 15, nanoparticles containing an oxide of the metal element (x1), nanoparticles containing a nitride of the metal element (x1), nanoparticles containing a sulfide of the metal element (x1), nanoparticles containing a carbide of the metal element (x1), and nanoparticles containing a boride of the metal element (x1).

[3] The composition according to the item [1] or [2], in which the polymer (Y) is one or more kind selected from the group consisting of a polymer (Y1) that contains two or more kinds selected from the group consisting of a constitutional unit derived from the following monomer (ya), a constitutional unit derived from the following monomer (yb), and a constitutional unit derived from the following monomer (yc), and a polymer (Y2) that contains a constitutional unit derived from the following monomer (yb):

monomer (ya): an olefin having 12 to 30 carbon atoms,

monomer (yb): a vinyl monomer having a nitrogen-containing heterocyclic group, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms, and

monomer (yc): a vinyl monomer having a nitrogen-containing heterocyclic group, all hydrogen atoms of which are not substituted by a chain-like alkyl group having 12 to 30 carbon atoms.

[4] The composition according to the item [3], in which the polymer (Y1) contains the constitutional unit derived from the monomer (ya) and the constitutional unit derived from the following monomer (yc).

[5] The composition according to the item [3] or [4], in which the monomer (ya) contains a linear α -olefin having 12 to 30 carbon atoms.

[6] The composition according to any one of the items [1] to [5], in which the nitrogen-containing heterocyclic group is a monovalent group obtained by removing one hydrogen atom from a pyrrolidone ring.

[7] The composition according to any one of the items [1] to [6], in which the composition has a content ratio ((Y)/(X)) of the polymer (Y) and the metal based nanoparticles (X) in terms of mass ratio of 0.2 or more.

[8] A method for producing the composition according to any one of the items [1] to [7], including the following step (1),

step (1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and one or more kind of a liquid dispersion medium selected from the group consisting of an organic solvent and a lubricant base oil,
the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group.

[9] The composition according to any one of the items [1] to [7], in which the composition is the additive composition for a lubricating oil composition or the additive composition for a grease composition, and the dispersion medium is one or more kind selected from the group consisting of an organic solvent and a lubricant

base oil.

[10] The composition according to the item [9], in which the composition is used as a wear resistant agent.

[11] A lubricating oil composition containing metal based nanoparticles (X), a polymer (Y), and a lubricant base oil,

the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,
the metal based nanoparticles (X) and the polymer (Y) being dispersed in the lubricant base oil.

[12] The lubricating oil composition according to the item [11], in which the metal based nanoparticles (X) have an average particle diameter of less than 600 nm.

[13] A grease composition containing metal based nanoparticles (X), a polymer (Y), and grease,

the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,
the metal based nanoparticles (X) and the polymer (Y) being dispersed in the grease.

[14] The grease composition according to the item [13], in which the grease composition has a volume resistivity of less than $1.0 \times 10^{13} \Omega\text{cm}$.

[15] The grease composition according to the item [14], in which the grease composition is used for preventing electrolytic corrosion.

Examples

[0179] The present invention will be described more specifically with reference to examples below. However, the present invention is not limited to the examples.

1. Investigation on Dispersibility of Metal based Nanoparticles (X)

[0180] The influence of the polymer species on the dispersibility of the metal based nanoparticles (X) was investigated.

<Materials>

[0181] The details of the organic solvent, the lubricant base oil, the metal based nanoparticles (X), and the polymers used in the production of the additive composition for a lubricating oil composition (which may be hereinafter referred to as an "additive composition") and the lubricating oil composition in the section "1. Investigation on Dispersibility of Metal based Nanoparticles (X)" are described below.

(Organic Solvent)

[0182] n-Heptane was used. n-Heptane is abbreviated as "heptane" in the following description.

(Lubricant Base Oil)

[0183] A hydrocarbon based mineral oil (40°C kinematic viscosity: 17.8 mm²/s) was used.

[0184] The 40°C kinematic viscosity of the lubricant base oil was measured according to JIS K2283:2000.

(Precursor of Metal based Nanoparticles (X))

[0185] ZnO: zinc oxide nanoparticles (available from IoLiTec GmbH, product name: zinc oxide, No. NO-0011-HP, primary particle diameter: 20 nm)

(Polymer (Y))

[0186]

Polymer (Y1): Antaron V-220 (available from Ashland Japan, Ltd.)
Antaron V-220 is a copolymer of 1-eicosene and N-vinylpyrrolidone.
Polymer (Y2): Antaron V-216 (available from Ashland Japan, Ltd.)

Antaron V-216 is a polymer a compound in which one of the hydrogen atoms that do not bonded to the nitrogen atom constituting the N-vinylpyrrolidone (i.e., the hydrogen atoms bonded to the carbon atoms constituting the heterocyclic ring) is substituted by a hexadecyl group.

(Polymer (Y'))

[0187]

Polymer (Y'1): Malialim AWS-0851 (available from NOF Corporation, polymer polycarboxylic acid)

Polymer (Y'2): copolymer of methyl methacrylate, N-(3-dimethylaminopropyl)methacrylamide, and dodecyl methacrylate (which may be hereinafter referred to as "acrylate polymer")

Polymer (Y'3): Esleam AD-508E (available from NOF Corporation, polymer amine compound)

Polymer (Y'4): Solperse 76500 (available from Lubrisol Corporation, comb-shaped urethane based dispersant)

Polymer (Y'5): Emulgen A-90 (available from Kao Corporation, polyoxyethylene distyrenated phenyl ether)

Polymer (Y'6): Malialim AAB-0851 (available from NOF Corporation, polymer polycarboxylic acid)

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

[0188] The method for producing the additive composition for a lubricating oil composition (which may be hereinafter referred simply to as an "additive composition") and the lubricating oil composition in the section "1. Investigation on Dispersibility of Metal based Nanoparticles (X)" is shown below.

[0189] In the following production examples, the content of the metal based nanoparticles (X) in the additive composition was regulated to 1% by mass (based on the total amount of the additive composition), and the content of the polymer therein was regulated to 2% by mass (based on the total amount of the additive composition).

[0190] In the following production examples, the content of the metal based nanoparticles (X) in the lubricating oil composition was regulated to 1% by mass (based on the total amount of the lubricating oil composition), and the content of the polymer therein was regulated to 2% by mass (based on the total amount of the lubricating oil composition).

<Production Example 1-1>

(Production Example A1-1: Preparation of Additive Composition (A1-1))

[0191] In a zirconia vessel (capacity: 100 mL), 31.0 g of heptane, 0.32 g of ZnO, 0.64 g of the polymer (Y2), and 152 g of zirconia beads (1) (particle diameter: 0.05 mm) were placed, and a bead mill treatment was performed at 2,000 rpm for 1.5 hours. The bead mill treatment was performed under a room temperature (25°C) environment. Subsequently, the liquid obtained by the bead mill treatment was filtered through a metal mesh filter to remove the zirconia beads, resulting in an additive composition (A1-1).

(Production Example B1-1: Preparation of lubricating oil composition (B1-1))

[0192] A part of the additive composition (A1-1) was separated, to which the lubricant base oil was added and mixed therein to make finally the aforementioned contents of the metal based nanoparticles (X) and the polymer in the lubricating oil composition, and then heptane was vaporized, resulting in a lubricating oil composition (B1-1).

<Production Example 1-2>

(Production Example A1-2: Preparation of Additive Composition (A1-2))

[0193] An additive composition (A1-2) was obtained in the same manner as in Production Example A1-1 except that the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

(Production Example B1-2: Preparation of Lubricating Oil Composition (B1-2))

[0194] A lubricating oil composition (B1-2) was obtained in the same manner as in Production Example B1-1 except that the additive composition (A1-1) was changed to the additive composition (A1-2).

<Production Example 1-3>

(Production Example A1-3: Preparation of Additive Composition (A1-3))

[0195] An additive composition (A1-3) was obtained in the same manner as in Production Example A1-1 except that the polymer (Y2) was changed to the polymer (Y1).

(Production Example B1-3: Preparation of Lubricating Oil Composition (B1-3))

[0196] A lubricating oil composition (B1-3) was obtained in the same manner as in Production Example B1-1 except that the additive composition (A1-1) was changed to the additive composition (A1-3).

<Comparative Production Example 1-1>

(Comparative Production Example A'1-1: Preparation of Additive Composition (A'1-1))

[0197] An additive composition (A'1-1) was obtained in the same manner as in Production Example A1-1 except that the polymer (Y2) was changed to the polymer (Y'1), and the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

(Production Example B'1-1: Preparation of Lubricating Oil Composition (B'1-1))

[0198] A lubricating oil composition (B'1-1) was obtained in the same manner as in Production Example B1-1 except that the additive composition (A1-1) was changed to the additive composition (A'1-1).

<Comparative Production Example 1-2>

(Comparative Production Example A'1-2: Preparation of Additive Composition (A'1-2))

[0199] An additive composition (A'1-2) was obtained in the same manner as in Production Example A1-1 except that the polymer (Y2) was changed to the polymer (Y'2), and the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

(Production Example B'1-2: Preparation of Lubricating Oil Composition (B'1-2))

[0200] A lubricating oil composition (B'1-2) was obtained in the same manner as in Production Example B1-1 except that the additive composition (A1-1) was changed to the additive composition (A'1-2).

<Comparative Production Example 1-3>

(Comparative Production Example A'1-3: Preparation of Additive Composition (A'1-3))

[0201] An additive composition (A'1-3) was obtained in the same manner as in Production Example A1-1 except that the polymer (Y2) was changed to the polymer (Y'3), and the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

<Comparative Production Example 1-4>

(Comparative Production Example A'1-4: Preparation of Additive Composition (A'1-4))

[0202] An additive composition (A'1-4) was obtained in the same manner as in Production Example A1-1 except that the polymer (Y2) was changed to the polymer (Y'4), and the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

<Comparative Production Example 1-5>

(Comparative Production Example A'1-5: Preparation of Additive Composition (A'1-5))

[0203] An additive composition (A'1-5) was obtained in the same manner as in Production Example A1-1 except that the polymer (Y2) was changed to the polymer (Y'5), and the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

<Comparative Production Example 1-6>

(Comparative Production Example A'1-6: Preparation of Additive Composition (A'1-6))

[0204] An additive composition (A'1-6) was obtained in the same manner as in Production Example A1-1 except that the polymer (Y2) was changed to the polymer (Y'6), and the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

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

[0205] The additive compositions and the lubricating oil compositions obtained in Production Examples above were investigated as follows.

<Investigation on Dispersibility 1: Investigation on Dispersion State in Additive Composition>

[0206] The dispersion state of the metal based nanoparticles (X) in the additive composition (in heptane) obtained in each of Production Examples A1-1 to A1-3 and Production Examples A'1-1 to A'1-6 was visually confirmed and evaluated by the following standard.

Evaluation A: No deposition was found with significantly good dispersibility.

Evaluation B: Slight deposition was found with good dispersibility.

Evaluation C: Deposition was found with poor dispersibility.

[0207] In the "Investigation on Dispersibility 1", the additive composition with the evaluation A or B was considered as acceptable.

[0208] For the additive composition with the evaluation A or B, the average particle diameter of the metal based nanoparticles (X) in the additive composition (in heptane) was measured. In Examples, the average particle diameter of the metal based nanoparticles (X) dispersed in the dispersion medium was measured by the dynamic light scattering method with Zetasizer Nano ZS, available from Malvern Panalytical Ltd. The measurement temperature was 25°C. The "average particle diameter" means an integrated value, Z-average particle diameter, in a particle size distribution obtained by the dynamic light scattering method, as described above.

<Investigation on Dispersibility 2: Investigation on Long-term Stability of Dispersion State in Lubricating Oil Composition>

[0209] The lubricating oil compositions (B1-1) to (B1-3), (B'1-1), and (B'1-2) each were stored in a transparent vessel and allowed to stand at room temperature (25°C), and the long-term stability of the dispersion state of the metal based nanoparticles (X) in the lubricating oil composition (in the lubricant base oil) was evaluated by the following standard.

Evaluation A: No deposition was found over 30 days or more.

Evaluation B: No deposition was found over 2 days or more, but deposition was found before the elapse of 30 days.

Evaluation C: Deposition was found before the elapse of 2 days.

[0210] In the "Investigation on Dispersibility 2", the lubricating oil composition with the evaluation A was considered as acceptable.

<Investigation on Dispersibility 3: Investigation on Dispersion State under High Temperature Environment>

[0211] The lubricating oil compositions (B1-1) to (B1-3) and (B'1-2) each were subjected to a thin film heating test, and the dispersion state of the metal based nanoparticles (X) after heating was investigated.

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[0212] The thin film heating test was performed in such a manner that 3 cc of the lubricating oil composition was placed in a glass cylindrical vessel having an inner diameter of 5 cm, and the lubricating oil composition in the form of thin film liquid was heated in a thermostat chamber in air under the following condition (1) or (2).

Condition (1): heating to 220°C for 12 hours

Condition (2): heating to 220°C for 3 hours, and then heating to 250°C for 1 hour

[0213] The occurrence of deposition in the lubricating oil composition after the thin film heating test was visually confirmed, and a case where no deposition was found was designated as the evaluation A, whereas a case where deposition was found was designated as the evaluation B.

[0214] The lubricating oil composition with the evaluation A causing no deposition was measured for the average particle diameter of the metal based nanoparticles (X) in the lubricating oil composition. The average particle diameter of the metal based nanoparticles (X) in the lubricating oil composition was measured in the same manner as described in the "Investigation on Dispersibility 1".

[0215] In the "Investigation on Dispersibility 3", the case with the evaluation A under both the conditions (1) and (2) was considered as acceptable.

[0216] The results are shown in Tables 1 and 2.

Table 1

		Example 1-1	Example 1-2	Example 1-3
Additive composition		(A1-1)	(A1-2)	(A1-3)
Lubricating oil composition		(B1-1)	(B1-2)	(B1-3)
Metal based nanoparticles (X)		ZnO	ZnO	ZnO
Polymer species		(Y2)	(Y2)	(Y1)
Details of polymer species		Antaron V-216	Antaron V-216	Antaron V-220
Average particle diameter of metal based nanoparticles (X) in additive composition (in heptane)		53 nm	100 nm	56 nm
Investigation on dispersibility 1 Dispersion state in additive composition (in heptane)		A	A	A
Investigation on dispersibility 2 Long-term stability of dispersion state in lubricating oil composition (in lubricant base oil)		A	A	A
Investigation on dispersibility 3 Thin film heating test *1	Condition (1)	A (300 nm)	A (350 nm)	A (300 nm)
	Condition (2)	A (350 nm)	A (350 nm)	A (350 nm)
*1: The numeral in parentheses is the average particle diameter of the metal based nanoparticles (X) in the lubricating oil composition (in the lubricant base oil).				

Table 2

	Comparative Example 1-1	Comparative Example 1-2	Comparative Example 1-3	Comparative Example 1-4	Comparative Example 1-5	Comparative Example 1-6
Additive composition	(A'1-1)	(A'1-2)	(A'1-3)	(A'1-4)	(A'1-5)	(A'1-6)
Lubricating oil composition	(B1'-1)	(B1'-2)	-	-	-	-
Metal based nanoparticles (X)	ZnO	ZnO	ZnO	ZnO	ZnO	ZnO
Polymer species	(Y'1)	(Y'2)	(Y'3)	(Y'4)	(Y'5)	(Y'6)
Details of polymer species	Malialim AWS-0851	acrylate polymer	Esleam AD-508E	Solsperse 76500	Emulgen A-90	Malialim AAB-0851
Average particle diameter of metal based nanoparticles (X) in additive composition (in heptane)	160 nm	100 nm	-	-	-	-
Investigation on dispersibility 1 Dispersion state in additive composition (in heptane)	B	A	C	C	C	C
Investigation on dispersibility 2 Long-term stability of dispersion state in lubricating oil composition (in lubricant base oil)	B	A	-	-	-	-
Investigation on dispersibility 3 Thin film heating test *1	-	A (1000 nm)	-	-	-	-
	-	B	-	-	-	-
*1: The numeral in parentheses is the average particle diameter of the metal based nanoparticles (X) in the lubricating oil composition (in the lubricant base oil).						

[0217] The following can be understood from the results shown in Tables 1 and 2.

[0218] Examples 1-1 to 1-3 using the polymer (Y1) or (Y2) satisfy the acceptance level in all the "Investigation on Dispersibility 1", the "Investigation on Dispersibility 2", and the "Investigation on Dispersibility 3".

[0219] On the other hand, Comparative Examples 1-3 to 1-6 using the polymers (Y'3) to (Y'6) do not reach the acceptance level in the "Investigation on Dispersibility 1".

[0220] Comparative Example 1-1 using the polymer (Y'1) satisfies the acceptance level in the "Investigation on Dispersibility 1", but does not reach the acceptance level in the "Investigation on Dispersibility 2".

[0221] Comparative Example 1-2 using the polymer (Y'2) satisfies the acceptance level in the "Investigation on Dispersibility 1" and the "Investigation on Dispersibility 2", but does not reach the acceptance level in the "Investigation on Dispersibility 3". It is understood from the measurement result of the average particle diameter of the metal based nanoparticles (X) in the thin film heating test under the condition (1) of Comparative Example 1-2 that with the use of the acrylate polymer in heating to a high temperature, the metal based nanoparticles (X) tend to aggregate due to the deteriorated dispersibility, failing to retain the small particle diameter.

[0222] It is understood from these results that the use of the polymer (Y1) or (Y2) can favorably disperse the metal based nanoparticles (X) in the dispersion medium, and can stably disperse the metal based nanoparticles (X) in the dispersion medium for a long time even under the high temperature environment expected in the use of a lubricating oil composition.

2. Investigation on Wear Resistance (1)

[0223] The lubricating oil compositions having the metal based nanoparticles (X) dispersed therein were investigated for the wear resistance.

[Examples 2-1 and 2-2 and Comparative Example 2-1]

[0224] The lubricating oil compositions (B1-1), (B1-2), and (B'1-2) produced in the section "1. Investigation on Dispersibility of Metal based Nanoparticles (X)" were subjected to a wear test according to the method described below.

<Wear Test>

[0225] In a high frequency friction machine, TE77, available from Phoenix Tribology Ltd., the lubricating oil composition was introduced between the test plate and the test ball, and the test was performed by moving the test ball under the following condition. The wear track diameter in the longitudinal direction and the wear track diameter in the transverse direction of the test ball after the test were measured, and the average value of the wear track diameters was calculated by the following expression.

Test plate: material: SUJ2, shape: length 58 mm × width 38 mm × thickness 3.9 mm

Test ball: material: SUJ2, diameter: 10 mm

Oil feed condition: oil bath, oil amount: 3 mL

Load: 50 N (300 seconds) → 100 N (300 seconds) → 150 N (300 seconds) → 200 N (300 seconds)

Temperature: 100°C

Amplitude: 10 mm

Frequency: 10 Hz

(average value of wear track diameter) = ((wear track diameter in longitudinal direction) + (wear track diameter in transverse direction)) / 2

[0226] It can be understood that the smaller the value of the wear track diameter is, the better the wear resistance of the lubricating oil composition is.

[0227] The wear test was performed under the following conditions (1) and (2).

Condition (1): The unheated lubricating oil composition was used.

Condition (2): The lubricating oil composition after heating to 220°C for 12 hours was used.

[0228] The results are shown in Table 3.

Table 3

		Example 2-1	Example 2-2	Comparative Example 2-1
5	Lubricating oil composition	(B1-1)	(B1-2)	(B'1-2)
	Metal based nanoparticles (X)	ZnO	ZnO	ZnO
	Polymer species	(Y2)	(Y2)	(Y'2)
10	Details of polymer species	Antaron V-216	Antaron V-216	Acrylate polymer
	Wear track diameter (average) (unit: μm)	Condition (1): not heated	412.44	421.28
		Condition (2): heated	460.72	463.17
				466.16
				651.98

[0229] The following can be understood from the results shown in Table 3.

[0230] It is understood that Examples 2-1 and 2-2 using the polymer (Y2) each exhibit a small wear track diameter under both the conditions (1) and (2), showing excellent wear resistance.

[0231] On the other hand, it is understood that Comparative Example 2-1 using the polymer (Y'2) exhibits a large wear track diameter under the condition (2). It is considered that the result is obtained since the metal based nanoparticles (X) tend to aggregate by heating the lubricating oil composition, largely deteriorating the effect of imparting the wear resistance by the metal based nanoparticles (X).

3. Investigation on Wear Resistance (2)

[0232] Subsequent to the section "2. Investigation on Wear Resistance (1)", the lubricating oil compositions having the metal based nanoparticles (X) dispersed therein were further investigated for the wear resistance.

<Materials>

[0233] The details of the organic solvent, the lubricant base oil, the metal based nanoparticles (X), and the polymers used in the production of the lubricating oil composition in the "3. Investigation on Wear Resistance (2)" are described below.

(Organic Solvent)

[0234] Heptane was used.

(Lubricant Base Oil)

[0235] A hydrocarbon based mineral oil (40°C kinematic viscosity: 17.8 mm²/s) was used, as similar to the section "1. Investigation on Dispersibility of Metal based Nanoparticles (X)".

(Precursor of Metal based Nanoparticles (X))

[0236]

ZnO: zinc oxide nanoparticles (available from IoLiTec GmbH, product name: zinc oxide, No. NO-0011-HP)

ZrO₂ (1): zirconia nanoparticles (available from Kanto Denka Kogyo Co., Ltd., product name: zirconia particles, Lot No.: 210125-011, primary particle diameter: 8 nm)

ZrO₂ (2): zirconia nanoparticles (available from Sigma-Aldrich Corporation, product name: zirconium oxide nano-powder, No. 544760, primary particle diameter: 100 nm)

WS₂: tungsten disulfide nanoparticles (available from IoLiTec GmbH, supplier code: NC-0016-HP, primary particle diameter: 90 nm, purity: 99%)

(Polymer (Y))

[0237]

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Polymer (Y1): Antaron V-220 (available from Ashland Japan, Ltd.)

Polymer (Y2): Antaron V-216 (available from Ashland Japan, Ltd.)

[Production Examples 3-1 to 3-8 and Comparative Production Examples 3-1 to 3-3]

[0238] The method for producing the lubricating oil composition in the section "3. Investigation on Wear Resistance (2)" is shown below.

(Production Example 3-1: Preparation of Lubricating Oil Composition (B3-1))

[0239] In a zirconia vessel (capacity: 100 mL), 31.0 g of the lubricant base oil, 0.32 g of ZnO, 0.64 g of the polymer (Y2), and 152 g of zirconia beads (1) (particle diameter: 0.05 mm) were placed, and a bead mill treatment was performed at 2,000 rpm for 1.5 hours. The bead mill treatment was performed under a room temperature (25°C) environment. Subsequently, the liquid obtained by the bead mill treatment was filtered through a metal mesh filter to remove the zirconia beads, resulting in a lubricating oil composition (B3-1).

(Production Example 3-2: Preparation of Lubricating Oil Composition (B3-2))

[0240] A lubricating oil composition (B3-2) was obtained in the same manner as in Production Example 3-1 except that the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

(Production Example 3-3: Preparation of Lubricating Oil Composition (B3-3))

[0241] In a zirconia vessel (capacity: 100 mL), 31.0 g of heptane, 0.32 g of ZrO_2 (1), 0.64 g of the polymer (Y2), and 152 g of zirconia beads (1) (particle diameter: 0.05 mm) were placed, and a bead mill treatment was performed at 2,000 rpm for 1.5 hours. The bead mill treatment was performed under a room temperature (25°C) environment. Subsequently, the liquid obtained by the bead mill treatment was filtered through a metal mesh filter to remove the zirconia beads, resulting in an additive composition (A3-3).

[0242] Subsequently, a part of the additive composition (A3-3) was separated, to which the lubricant base oil was added and mixed therein to make finally the contents of the metal based nanoparticles (X) and the polymer in the lubricating oil composition shown in Table 4, and then heptane was vaporized, resulting in a lubricating oil composition (B3-3).

(Production Example 3-4: Preparation of Lubricating Oil Composition (B3-4))

[0243] A lubricating oil composition (B3-4) was obtained in the same manner as in Production Example 3-3 except that the polymer (Y2) was changed to the polymer (Y1).

(Production Example 3-5: Preparation of Lubricating Oil Composition (B3-5))

[0244] A lubricating oil composition (B3-5) was obtained in the same manner as in Production Example 3-4 except that the amount of the polymer (Y1) added was changed to 0.32 g.

(Production Example 3-6: Preparation of Lubricating Oil Composition (B3-6))

[0245] A lubricating oil composition (B3-6) was obtained in the same manner as in Production Example 3-4 except that the zirconia beads (1) were changed to 152 g of zirconia beads (2) (particle diameter: 0.10 mm).

(Production Example 3-7: Preparation of Lubricating Oil Composition (B3-7))

[0246] A lubricating oil composition (B3-7) was obtained in the same manner as in Production Example 3-4 except that ZrO_2 (1) was changed to ZrO_2 (2), the amount of the polymer (Y1) added was changed to 0.13 g, and the zirconia beads (1) were changed to 152 g of zirconia beads (3) (particle diameter: 0.20 mm).

(Production Example 3-8: Preparation of Lubricating Oil Composition (B3-8))

[0247] A lubricating oil composition (B3-8) was obtained in the same manner as in Production Example 3-3 except that ZnO (1) was changed to WS_2 , and the zirconia beads (1) were changed to 152 g of zirconia beads (3) (particle diameter: 0.20 mm).

(Comparative Production Example 3-1: Preparation of Lubricating Oil Composition (B'3-1))

[0248] 99% by mass of the lubricant base oil and 1% by mass of the polymer (Y1) were mixed to provide a lubricating oil composition (B'3-1).

(Comparative Production Example 3-2: Preparation of Lubricating Oil Composition (B'3-2))

[0249] 99.39% by mass of the lubricant base oil and 0.61% by mass of zinc dialkyldithiophosphate (ZnDTP) were mixed to provide a lubricating oil composition (B'3-2).

(Comparative Production Example 3-3: Preparation of Lubricating Oil Composition (B'3-3))

[0250] 99.95% by mass of the lubricant base oil and 0.05% by mass of zirconium complex were mixed to provide a lubricating oil composition (B'3-3).

[Examples 3-1 to 3-8 and Comparative Examples 3-1 to 3-4]

[0251] The lubricating oil compositions obtained in Production Examples and the lubricant base oil each were measured for the wear track diameter by subjecting to the same wear test as in the section "2. Investigation on Wear Resistance (1)" (only under the condition (1)), and the case with a wear track diameter (average) of 470 μm or less was considered as acceptable.

[0252] The average particle diameter of the metal based nanoparticles (X) in the lubricating oil composition (in the lubricant base oil) was measured in the same manner as in the measurement method described in the section "1. Investigation on Dispersibility of Metal based Nanoparticles (X)".

[0253] The results are shown in Tables 4 and 5.

Table 4

	Example 3-1	Example 3-2	Example 3-3	Example 3-4	Example 3-5	Example 3-6	Example 3-7	Example 3-8
Kind of lubricating oil composition	(B3-1)	(B3-2)	(B3-3)	(B3-4)	(B3-5)	(B3-6)	(B3-7)	(B3-8)
Metal based nanoparticles (X)	ZnO	ZnO	ZrO ₂ (1)	ZrO ₂ (1)	ZrO ₂ (1)	ZrO ₂ (1)	ZrO ₂ (2)	WS ₂
Concentration of metal based nanoparticles (X) (% by mass)	1	1	1	1	1	1	1	1
Concentration of polymer (% by mass)	2	2	2	2	1	2	0.4	2
Polymer species	(Y2)	(Y2)	(Y2)	(Y1)	(Y1)	(Y1)	(Y1)	(Y2)
Details of polymer species	Antaron V-216	Antaron V-216	Antaron V-216	Antaron V-220	Antaron V-220	Antaron V-220	Antaron V-220	Antaron V-216
Average particle diameter of metal based nanoparticles (X)	60 nm	100nm	55nm	45nm	70nm	100nm	200nm	180nm

(continued)

	Example 3-1	Example 3-2	Example 3-3	Example 3-4	Example 3-5	Example 3-6	Example 3-7	Example 3-8
Wear track diameter (longitudinal) (unit: μm)	389.6	406.4	404.84	402.76	376.5	405.06	383.63	376.46
Wear track diameter (transverse) (unit: μm)	419.5	418.29	417.12	415.91	400.37	416.97	459.02	393.19
Wear track diameter (average) (unit: μm)	404.55	412.35	410.98	409.34	388.44	411.02	421.325	384.825

Table 5

	Comparative Example 3-1	Comparative Example 3-2	Comparative Example 3-3	Comparative Example 3-4
Kind of lubricating oil composition	base oil only	(B'3-1)	(B'3-2)	(B'3-3)
Metal based compound	-	-	ZnDTP	zirconium complex
Concentration of metal based compound (% by mass)	0	0	0.61	0.05
Concentration of polymer (% by mass)	0	0.4	-	-
Polymer species	-	(Y1)	-	-
Details of polymer species	-	Antaron V-220	-	-
Average particle diameter	-	-	-	-
Wear track diameter (longitudinal) (unit: μm)	645.36	522.26	472.07	458.92
Wear track diameter (transverse) (unit: μm)	658.59	511.51	506.81	484.03
Wear track diameter (average) (unit: μm)	651.975	516.885	489.44	471.48

[0254] The following can be understood from Tables 4 and 5.

[0255] It is understood that Examples 3-1 to 3-8 each exhibit a wear track diameter (average) of 470 μm or less, showing excellent wear resistance.

[0256] On the other hand, it is understood that Comparative Examples 3-1 to 3-4 each exhibit a wear track diameter (average) exceeding 470 μm , showing inferior wear resistance.

[0257] The ZnDTP and the zirconium complex used in Comparative Examples 3-3 and 3-4 are wear resistant agents that have been ordinarily blended in lubricating oil compositions, and it is understood that Examples provide wear resistance better than the wear resistant agents.

4. Investigation on Grease Composition

[0258] The effect of the addition of an additive composition for a grease composition (which may be hereinafter referred simply to as an "additive composition") to a grease composition was variously investigated.

<Materials>

[0259] The details of the organic solvent, the lubricant base oil, the metal based nanoparticles (X), the polymers, and the grease used in the production of the grease composition in the section "4. Investigation on grease composition" are described below.

(Organic Solvent)

[0260] Heptane was used.

(Lubricant Base Oil)

[0261] An alkylated diphenyl ether (Moresco Hilube LB-100, available from Moresco Corporation) was used.

(Precursor of Metal based Nanoparticles (X))

[0262]

TiN: titanium nitride nanoparticles (available from EM Japan Co., Ltd., primary particle diameter: 20 nm, purity: 99.2% or more)

WS₂: tungsten disulfide nanoparticles (available from IoLiTec GmbH, supplier code: NC-0016-HP, primary particle diameter: 90 nm, purity: 99%)

(Metal based Powder (X'))

[0263]

Powder TiN: titanium nitride particles having an average particle diameter of 2 μm

Powder WS₂: tungsten disulfide particles having an average particle diameter of 2 μm

Carbon black: carbon particles having an average particle diameter of 2 μm

(Polymer (Y))

[0264] Polymer (Y2): Antaron V-216 (available from Ashland Japan, Ltd.)

(Polymer (Y'))

[0265] Polymer (Y'2): acrylate polymer

(Base Grease)

[0266] Base grease used included an alkylated diphenyl ether as a base oil (the same base oil as the lubricant base oil above) and an aliphatic diurea as a thickening agent. The mixing ratio of the thickening agent and the base oil was 1/5 in terms of mass ratio.

[0267] The aliphatic diurea was an aliphatic diurea that was synthesized from octylamine and diphenylmethane diisocyanate.

[Production Examples 4-1 and 4-2 and Comparative Production Examples 4-1 to 4-4]

[0268] The method for producing the grease composition in the section "4. Investigation on grease composition" is shown below.

<Production Example 4-1: Preparation of Grease Composition (C4-1)>

(Production Example A4-1: Preparation of Additive Composition (A4-1))

[0269] In a zirconia vessel (capacity: 100 mL), 17.6 g of heptane, 4.8 g of TiN, 9.6 g of the polymer (Y2), and 152 g of zirconia beads (particle diameter: 0.05 mm) were placed, and a bead mill treatment was performed at 2,000 rpm for 1.5

hours. The bead mill treatment was performed under a room temperature (25°C) environment. Subsequently, the liquid obtained by the bead mill treatment was filtered through a metal mesh filter to remove the zirconia beads.

[0270] Subsequently, the lubricant base oil was mixed with the liquid obtained by removing the zirconia beads, from which pentane was vaporized, resulting in an additive composition (A4-1) having a content of the metal based nanoparticles (X) of 20% by mass.

(Production Example C4-1: Preparation of Grease Composition (C4-1))

[0271] The base grease and the additive composition (A4-1) were mixed to make a content of the metal based nanoparticles (X) in the grease composition of 4% by mass based on the total amount of the grease composition, resulting in a grease composition (C4-1).

<Production Example 4-2: Preparation of Grease Composition (C4-2)>

(Production Example A4-2: Preparation of Additive Composition (A4-2))

[0272] An additive composition (A4-2) was obtained in the same manner as in Production Example A4-1 except that TiN was changed to WS₂, and the zirconia beads (1) were changed to 152 g of zirconia beads (3) (particle diameter: 0.20 mm)

(Production Example C4-2: Preparation of Grease Composition (C4-2))

[0273] A grease composition (C4-2) was obtained in the same manner as in Production Example C4-1 except that the additive composition (A4-1) was changed to the additive composition (A4-2).

<Comparative Production Example 4-1: Preparation of Grease Composition (C'4-1)>

[0274] The powder TiN and the lubricant base oil were mixed to provide a mixed liquid, and the mixed liquid and the base grease were mixed to make a content of the powder TiN in the grease composition of 4% by mass based on the total amount of the grease composition, from which pentane was vaporized, resulting in a grease composition (C'4-1).

<Comparative Production Example 4-2: Preparation of Grease Composition (C'4-2)>

[0275] The powder WS₂ and the lubricant base oil were mixed to provide a mixed liquid, and the mixed liquid and the base grease were mixed to make a content of the powder WS₂ in the grease composition of 4% by mass based on the total amount of the grease composition, from which pentane was vaporized, resulting in a grease composition (C'4-2).

<Comparative Production Example 4-3: Preparation of Grease Composition (C'4-3)>

[0276] The carbon and the lubricant base oil were mixed to provide a mixed liquid, and the mixed liquid and the base grease were mixed to make a content of the carbon in the grease composition of 4% by mass based on the total amount of the grease composition, from which pentane was vaporized, resulting in a grease composition (C'4-3).

<Comparative Production Example 4-4: Preparation of Grease Composition (C'4-4)>

(Comparative Production Example A'4-4: Preparation of Additive Composition (A'4-4))

[0277] An additive composition (A'4-4) was obtained in the same manner as in Production Example A4-1 except that the polymer (Y2) was changed to the polymer (Y'2).

(Comparative Production Example C'4-4: Preparation of Grease Composition (C'4-4))

[0278] A grease composition (C'4-4) was obtained in the same manner as in Production Example C4-1 except that the additive composition (A4-1) was changed to the additive composition (A'4-4).

[Examples 4-1 and 4-2 and Comparative Examples 4-1 to 4-5]

[0279] The grease compositions produced in Production Examples above and the base grease were investigated as follows.

<Evaluation of Volume Resistivity>

[0280] The volume resistivity was measured under the following condition with a digital ultrahigh resistance-micro-current meter (ADCMT5451) and a test fixture for volume resistivity (ADCMT12707), all available from ADC Corporation, using a dedicated electrode for 12707.

Measurement voltage: 40 V

Sample amount: 0.8 g

<Wear Test>

[0281] In a high frequency friction machine, TE77, available from Phoenix Tribology Ltd., the grease composition was introduced between the test plate and the test ball, and the test was performed by moving the test ball under the following condition. The wear track diameter in the longitudinal direction and the wear track diameter in the transverse direction of the test ball after the test were measured, and the average value of the wear track diameters was calculated by the following expression.

Test plate: material: SUJ2, shape: length 58 mm × width 38 mm × thickness 3.9 mm

Test ball: material: SUJ2, diameter: 10 mm

Grease feed condition: grease bath, grease amount: 3 mL

Load: 50 N (300 seconds)

Temperature: 100°C

Amplitude: 10 mm

Frequency: 10 Hz

$$(\text{average value of wear track diameter}) = ((\text{wear track diameter in longitudinal direction}) + (\text{wear track diameter in transverse direction})) / 2$$

[0282] It can be understood that the smaller the value of the wear track diameter is, the better the wear resistance of the grease composition is.

[0283] It can be also understood that the smaller the value of the friction coefficient is, the better the friction characteristics of the grease composition is.

[0284] The results are shown in Table 6.

[0285] Table 6 also shows the measurement results of the average particle diameter of the metal based nanoparticles (X) dispersed in heptane in Production Example 4-1, Production Example 4-2, and Comparative Production Example 4-4. The average particle diameter was measured in the same manner as in the section "1. Investigation on Dispersibility of Metal based Nanoparticles (X)".

Table 6

	Example 4-1	Example 4-2	Comparative Example 4-1	Comparative Example 4-2	Comparative Example 4-3	Comparative Example 4-4	Comparative Example 4-5
Grease composition	(C4-1)	(C4-2)	(C'4-1)	(C'4-2)	(C'4-3)	(C'4-4)	base grease
Additive composition	(A4-1)	(A4-2)	-	-	-	(A'4-4)	-
Metal based nanoparticles (X)	TiN	WS ₂	-	-	-	TiN	-
Metal based particles (X')	-	-	powder TiN	powder WS ₂	carbon black	-	-
Polymer species	(Y1)	(Y1)	-	-	-	(Y'2)	-
Detail of polymer species	Antaron V-216	Antaron V-216	-	-	-	acrylate polymer	-

(continued)

		Example 4-1	Example 4-2	Comparative Example 4-1	Comparative Example 4-2	Comparative Example 4-3	Comparative Example 4-4	Comparative Example 4-5
5	Average particle diameter of metal based nano-particles (X) in additive composition (heptane)	62 nm	180 nm	-	-	-	300 nm	-
10								
15								
20								
	Volume resistivity (Ωcm)	7.23 × 10 ¹¹	9.98 × 10 ¹¹	5.32 × 10 ¹⁴	5.98 × 10 ¹⁴	6.02 × 10 ¹⁴	4.81 × 10 ¹⁴	3.52 × 10 ¹⁵
	Wear track diameter (μm)	230.66	215.72	567.675	282.065	540.79	530.09	403.355
	Friction coefficient (maximum value)	0.065	0.060	0.337	0.075	0.38	0.366	0.314

[0286] The following can be understood from Table 6.

[0287] It is understood that the grease compositions of Examples 4-1 and 4-2 each have a high electroconductivity and are excellent in friction and wear characteristics.

[0288] On the other hand, the grease compositions of Comparative Examples 4-1 to 4-5 each have a low electroconductivity and are inferior in friction and wear characteristics.

[0289] It is considered from the comparison between Example 4-1 and Comparative Example 4-4 that the grease composition of Example has a low volume resistivity and is excellent in wear resistance as a result of the high-level dispersion of the metal based nanoparticles (X).

Claims

1. A composition comprising metal based nanoparticles (X), a polymer (Y), and a dispersion medium,

the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,

the metal based nanoparticles (X) and the polymer (Y) being dispersed in the dispersion medium,

the composition being used as an additive composition for a lubricating oil composition, an additive composition for a grease composition, a lubricating oil composition, or a grease composition.

2. The composition according to claim 1, wherein the metal based nanoparticles (X) contains one or more kind of metal based nanoparticles (X1) selected from the group consisting of metal nanoparticles containing one or more kind of a metal element (x1) selected from the group consisting of transition metal elements, and metal elements and semimetal elements of Groups 12 to 15, nanoparticles containing an oxide of the metal element (x1), nanoparticles containing a nitride of the metal element (x1), nanoparticles containing a sulfide of the metal element (x1), nanoparticles containing a carbide of the metal element (x1), and nanoparticles containing a boride of the metal element (x1).

3. The composition according to claim 1 or 2, wherein the polymer (Y) is one or more kind selected from the group consisting of a polymer (Y1) that contains two or more kinds selected from the group consisting of a constitutional unit derived from the following monomer (ya), a constitutional unit derived from the following monomer (yb), and a constitutional unit derived from the following monomer (yc), and a polymer (Y2) that contains a constitutional unit derived from the following monomer (yb) and does not contain a constitutional unit derived from the following monomer (ya) and a constitutional unit derived from the following monomer (yc):

monomer (ya): an olefin having 12 to 30 carbon atoms,

monomer (yb): a vinyl monomer having a nitrogen-containing heterocyclic group, at least one hydrogen atom of which is substituted by a chain-like alkyl group having 12 to 30 carbon atoms, and
 monomer (yc): a vinyl monomer having a nitrogen-containing heterocyclic group, all hydrogen atoms of which are not substituted by a chain-like alkyl group having 12 to 30 carbon atoms.

4. The composition according to claim 3, wherein the polymer (Y1) contains the constitutional unit derived from the monomer (ya) and the constitutional unit derived from the following monomer (yc).
5. The composition according to claim 3 or 4, wherein the monomer (ya) contains a linear α -olefin having 12 to 30 carbon atoms.
6. The composition according to any one of claims 1 to 5, wherein the nitrogen-containing heterocyclic group is a monovalent group obtained by removing one hydrogen atom from a pyrrolidone ring.
7. The composition according to any one of claims 1 to 6, wherein the composition has a content ratio ((Y)/(X)) of the polymer (Y) and the metal based nanoparticles (X) in terms of mass ratio of 0.2 or more.
8. A method for producing the composition according to any one of claims 1 to 7, comprising the following step (1),
 step (1): a step of performing a dispersing treatment of a precursor of metal based nanoparticles (X) in the presence of a polymer (Y) and one or more kind of a liquid dispersion medium selected from the group consisting of an organic solvent and a lubricant base oil,
 the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group.
9. The composition according to any one of claims 1 to 7, wherein the composition is the additive composition for a lubricating oil composition or the additive composition for a grease composition, and the dispersion medium is one or more kind selected from the group consisting of an organic solvent and a lubricant base oil.
10. The composition according to claim 9, wherein the composition is used as a wear resistant agent.
11. A lubricating oil composition comprising metal based nanoparticles (X), a polymer (Y), and a lubricant base oil,
 the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,
 the metal based nanoparticles (X) and the polymer (Y) being dispersed in the lubricant base oil.
12. The lubricating oil composition according to claim 11, wherein the metal based nanoparticles (X) have an average particle diameter of less than 600 nm.
13. A grease composition comprising metal based nanoparticles (X), a polymer (Y), and grease,
 the polymer (Y) being a vinyl polymer having a chain-like alkyl group having 10 to 28 carbon atoms and a nitrogen-containing heterocyclic group,
 the metal based nanoparticles (X) and the polymer (Y) being dispersed in the grease.
14. The grease composition according to claim 13, wherein the grease composition has a volume resistivity of less than $1.0 \times 10^{13} \Omega\text{cm}$.
15. The grease composition according to claim 14, wherein the grease composition is used for preventing electrolytic corrosion.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2023/013056

A. CLASSIFICATION OF SUBJECT MATTER

C10M 169/04(2006.01)i; **C10M 125/04**(2006.01)i; **C10M 125/08**(2006.01)i; **C10M 125/10**(2006.01)i;
C10M 125/26(2006.01)i; **C10M 149/02**(2006.01)i; **C10M 161/00**(2006.01)i; **C10N 10/04**(2006.01)n; **C10N 10/06**(2006.01)n;
C10N 10/08(2006.01)n; **C10N 10/10**(2006.01)n; **C10N 20/06**(2006.01)n; **C10N 40/02**(2006.01)n; **C10N 40/04**(2006.01)n;
C10N 40/06(2006.01)n; **C10N 40/08**(2006.01)n; **C10N 40/12**(2006.01)n; **C10N 40/25**(2006.01)n; **C10N 40/30**(2006.01)n;
C10N 50/10(2006.01)n
 FI: C10M169/04; C10M125/04; C10M125/08; C10M125/10; C10M125/26; C10M149/02; C10M161/00; C10N10/04;
 C10N10/06; C10N10/08; C10N10/10; C10N20/06; C10N40/02; C10N40/04; C10N40/06; C10N40/08; C10N40/12;
 C10N40/25; C10N40/30; C10N50/10

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)

C10M169/04; C10M125/04; C10M125/08; C10M125/10; C10M125/26; C10M149/02; C10M161/00; C10N10/04;
 C10N10/06; C10N10/08; C10N10/10; C10N20/06; C10N40/02; C10N40/04; C10N40/06; C10N40/08; C10N40/12;
 C10N40/25; C10N40/30; C10N50/10

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

Published examined utility model applications of Japan 1922-1996
 Published unexamined utility model applications of Japan 1971-2023
 Registered utility model specifications of Japan 1996-2023
 Published registered utility model applications of Japan 1994-2023

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2007-186698 A (NATIONAL STARCH AND CHEMICAL INVESTMENT HOLDING CORP.) 26 July 2007 (2007-07-26) claims, paragraph [0006]	1-15
X	JP 47-8279 A (AMERICAN METAL CLIMAX INC.) 01 May 1972 (1972-05-01) claims, p. 2, lower right column, last line to p. 3, upper left column, line 1, p. 6, lower right column, line 3 from the bottom to p. 7, upper left column, line 1, examples 1, 2	1-2, 6-15
X	JP 2003-503543 A (QUAKER CHEMICAL CORP.) 28 January 2003 (2003-01-28) example 4	11-12

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

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"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

02 June 2023

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2023/013056

C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2013-544328 A (EVONIK OIL ADDITIVES GMBH) 12 December 2013 (2013-12-12) in particular, examples 5-9, 14-22	1-15

Form PCT/ISA/210 (second sheet) (January 2015)

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/JP2023/013056

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
JP 2007-186698 A	26 July 2007	US 2007/0161518 A1 claims, paragraph [0006] EP 1808477 A1 CN 100999692 A KR 10-2007-0075309 A	
JP 47-8279 A	01 May 1972	US 3842009 A claims, examples 1, 2	
JP 2003-503543 A	28 January 2003	US 6548456 B1 example 4 WO 2000/078901 A1 EP 1063280 A1 CN 1357032 A KR 10-2002-0050759 A	
JP 2013-544328 A	12 December 2013	US 2013/0219868 A1 in particular, examples 5-9, 14-22 WO 2012/056022 A1 EP 2633011 A1 CN 103201362 A KR 10-2014-0066968 A	

Form PCT/ISA/210 (patent family annex) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

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- JP 2021512188 A [0004]
- JP 2021512189 A [0004]