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(54) **ANTI-FLAKING AGENT AND LUBRICANT COMPOSITION COMPRISING THE SAME**
ANTIFLOCKUNGSMITTEL UND SCHMIERMITTELZUSAMMENSETZUNG DAMIT
AGENT ANTI-ÉCAILLAGE ET COMPOSITION DE LUBRIFIANT LE COMPRENANT

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Description

[Technical Field]

5 **[0001]** The present invention relates to a novel anti-flaking agent which can be contained in a lubricant applicable to a metal surface of e.g. a rolling bearing. The present invention also relates to a lubricant composition containing the anti-flaking agent.

[Background Art]

10 **[0002]** A peculiar early abnormal flaking involving formation of white etching area occurring on the rolling surface of a rolling bearing has been a problem since the mid-1980s because it reduces the fatigue life of the rolling bearing. Such flaking is called white flaking, white band flaking, brittle flaking, hydrogen brittle flaking, or hydrogen embrittlement flaking.

15 **[0003]** Although the mechanism of how such flaking takes place has not yet been elucidated, Patent Literature 1 introduces a hydrogen hypothesis, for example. Specifically, the hypothesis is as follows: when grease is used under a high load, the grease decomposes to generate hydrogen; the hydrogen penetrates into the steel material of the rolling bearing and reacts with carbide at the grain boundaries; and as a result, the steel material becomes brittle. Patent Literature 1 reports that, when a grease composition contains a specific compound containing at least one sulfur atom such as a thiazole derivative, a sulfurized oil and/or fat, or a sulfurized olefin, it is possible to deal with the problem of white band flaking, that is, the intrusion of hydrogen generated by decomposition of the lubricant into the metal.

20 **[0004]** The mechanism of how flaking takes place is also explained from the viewpoint of the formation of a new metal surface. Specifically, the mechanism is as follows: when the metal transfer surface wears, a new surface is easily formed by the wear; the newly formed surface brings about catalysis to chemically decompose the grease; and as a result, a large amount of hydrogen is generated, and the generated hydrogen penetrates into the steel to finally produce cracks on the metal surface. Patent Literature 2 reports an additive which is a passivating oxidizer such as a nitrite, where the additive is added to the grease to oxidize the metal surface and suppress the catalytic activity of the surface, thereby suppressing the generation of hydrogen due to the decomposition of the lubricant. Patent Literature 3 reports a technique of combining a passivating oxidizer with an organic sulfonate. Patent Literature 4 reports a technique of allowing grease to contain a specific amount of an azo compound. Patent Literature 5 reports a technique that suppresses the generation of hydrogen from grease by using a phenyl ether-based synthetic oil as the base oil of the grease.

[Citation List]

[Patent Literature]

35 **[0005]**

[Patent Literature 1] International Publication No. WO2015/016376

[Patent Literature 2] Japanese Patent Application Publication No. Hei 3-210394

40 [Patent Literature 3] Japanese Patent Application Publication No. Hei 5-263091

[Patent Literature 4] Japanese Patent Application Publication No. 2002-130301

[Patent Literature 5] Japanese Patent Application Publication No. Hei 3-250094

45 **[0006]** The following patent literature also refers to grease and/or lubricant compositions. EP 3 029 132 A1; EP 2 003 187 A2; EP 2 239 313 A1; EP 3 336 161 A1; EP 2 431 449 A1; EP 1 609 844 A1; US 2009/069204 A1; CN 106 281 620 A; CN 107 312 589 A; US 2015/057203 A1; EP 1 516 911 A1; and US 5 462 683 A.

[Summary of Invention]

[Technical Problems]

50 **[0007]** Meanwhile, it is known that plasma is generated in a minute range of several μm to several mm on the friction surface (Nakayama, K., Yagasaki, F., Tribology Letters (2018)). Such plasma is called "triboplasma." Discharge luminescence and electric corrosion also take place on an elastohydrodynamic lubrication (EHL) thin film of grease formed on rolling bearings. From these facts, there is a report suggesting that discharge plasma is generated on an EHL thin film (Nakayama and Tanaka: Manuscript Preparation for Tribology Conference, Tokyo (2016) A2).

55 **[0008]** The present inventors considered that the suppression of triboplasma generation could prevent white band flaking of e.g. a rolling bearing.

[0009] In view of the above, an object of the present invention is to provide an anti-flaking agent capable of suppressing white band flaking of e.g. a rolling bearing, and a lubricant composition containing the anti-flaking agent.

[Solution to Problems]

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[0010] The present inventors measured the amount of hydrogen generated using a candidate compound for a base oil accounting for a large percentage of the lubricant composition or the grease composition, and have found that a compound having a specific volume resistivity of $1.0 \times 10^{10} \Omega \cdot \text{cm}$ or less can effectively suppress hydrogen generation. Based on this knowledge, the present inventors have completed an invention which can effectively prevent white band flaking of e.g. a rolling bearing.

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[0011] Specifically, the present invention provides the following use.

[0012]

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[1] Use as an anti-flaking agent of at least one selected from the group consisting of (A) a compound having a specific volume resistivity of $1.0 \times 10^{10} \Omega \cdot \text{cm}$ or less, wherein the compound (A) is a diester selected from the group consisting of dimethyl phthalate, dimethyl maleate, diethyl malonate, dibutyl malonate, and dihexyl malonate.

[0013] The present invention also provides the following lubricant composition.

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[2] A lubricant composition comprising a diester selected from the group consisting of dimethyl maleate, dibutyl malonate, and dihexyl malonate and least one conventional base oil selected from the group consisting of mineral oils and synthetic oils.

[3] The lubricant composition according to 2 described above, wherein the base oil is at least one selected from the group consisting of mineral oils, synthetic hydrocarbon oils, and ether oils.

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[4] The lubricant composition according to any one of 2 to 3 described above, wherein a content of the compound exceeds 0.1% by mass based on a total mass of the composition.

[5] The lubricant composition according to any one of 2 to 3 described above, wherein a content of the compound exceeds 1% by mass based on a total mass of the composition.

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[6] The lubricant composition according to any one of 2 to 3 described above, wherein a content of the compound is 2% by mass or more based on a total mass of the composition.

[7] The lubricant composition according to any one of 2 to 3 described above, wherein a content of the compound is 3% by mass or more based on a total mass of the composition.

[8] The lubricant composition according to any one of 2 to 3 described above, wherein a content of the compound is 40% by mass or less based on a total mass of the composition.

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[9] The lubricant composition according to any one of 2 to 8 described above, further comprising a thickener.

[0014] The anti-flaking agent and lubricant composition of the present invention can prevent white band flaking effectively (20% or less as compared with n-hexadecane).

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[Brief Description of Drawings]

[0015] [Fig. 1] Fig. 1 is a schematic view of an apparatus, used in Examples, for generating hydrogen gas by triboplasma.

[Description of Embodiments]

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[Definitions]

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[0016] In the present specification, the specific volume resistivity represents a ratio between a DC electric field (V/m) applied to the sample at 25°C and a current per unit cross-sectional area applied to the sample at that time, and is equal to the resistance between opposing faces of a cubic sample with one side being 1 cm. The specific volume resistivity can be measured based on the testing methods of electrical insulating oils specified in JIS C2101.

[0017] In the present specification, the dielectric constant ϵ is a coefficient representing the relationship between the electric charge in the substance and the force given thereby. The dielectric constant ϵ was measured at 25°C with E4991B Impedance Analyzer (Keysight Technologies).

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[0018] In the present specification, the "Hansen solubility parameters" are each an index indicating the solubility of a certain solute in a certain solvent, and include three components: the dispersion term (δD), the polar term (δP), and the hydrogen bond term (δH). The dispersion term (δD) represents the effect due to the dispersion force, the polar term (δP) represents the effect due to the force between dipoles, and the hydrogen bond term (δH) represents the effect due to the

hydrogen bond force. Details of the definitions and calculation methods for the Hansen solubility parameters are described in the following literature: Charles M. Hansen, "Hansen Solubility Parameters: A Users Handbook," CRC Press, 2007.

[0019] In the present specification, "white band flaking" refers to a peculiar early abnormal flaking involving formation of white etching area. In the present specification, the term "white band flaking" is synonymous with a term called e.g. white flaking, white band flaking, brittle flaking, hydrogen brittle flaking, or hydrogen embrittlement flaking in the art. Normally, for rolling fatigue, the life can be estimated based on the life calculation formula defined in the standards (ISO281, JIS B-1518). However, in the case where white band flaking takes place, the lifetime is reached in a shorter time than the calculated lifetime. In the actual market, it has been reported that the life is reached at about 1/10 to 1/20 of the calculated life. White band flaking is one type of internal origin damage, and shows a specific phenomenon in which a white band is observed when the metal structure after the occurrence is etched with a nital solution.

[Compound Used as Anti-Flaking Agent]

[0020] The compound used in the present invention is a compound having a specific volume resistivity of $1.0 \times 10^{10} \Omega \cdot \text{cm}$ or less, and is a diester as defined above. The present inventors have found that a compound having such a physical property can suppress hydrogen generation by plasma. Although the experimental methods and results are described in detail in the Examples section, the present inventors systematically examined the influence of the carbon chain length of ester on the amount of hydrogen generated. Then, as regards the length of the carbon chain derived from the dibasic fatty acid constituting the ester with methanol ($R^2\text{OOC-R}^1\text{-COOR}^2$) (that is, R^1), no hydrogen was generated when the number of carbon atoms was 6 or less. Conversely, when the number of carbon atoms was 8 (that is, when the dibasic acid was sebacic acid), hydrogen was generated. However, the amount generated was only 17% compared with n-hexadecane used as a standard substance. It was considered that triboplasma was generated in the case of using dimethyl sebacate. Thus, the specific volume resistivity was measured and found to be $9.0 \times 10^9 \Omega \cdot \text{cm}$. Moreover, when the specific volume resistivity was measured while changing the number of carbon atoms of R^1 , it was found that the specific volume resistivity increased as the number of carbon atoms of R^1 increased.

Table 1

	Number of Carbon Atoms of R^1	Amount of Hydrogen Generated, %*	Specific Volume Resistivity, $\Omega \cdot \text{cm}$
Dimethyl Malonate	1	0	6.6×10^7
Dimethyl Adipate	4	0	4.0×10^9
Dimethyl Sebacate	8	17	9.0×10^9
* The amount of hydrogen generated for n-hexadecane is set to 100.			

[0021] On the other hand, the present inventors examined the influence of the length of the carbon chain derived from the alcohol constituting the ester with sebacic acid (that is, R^2) on the amount of hydrogen generated. Then, it was found that the specific volume resistivity increased as the number of carbon atoms of R^2 increased. This tendency was also observed in the case of monoesters.

Table 2

	Number of Carbon Atoms of R^2	Amount of Hydrogen Generated, %*	Specific Volume Resistivity, $\Omega \cdot \text{cm}$
Dimethyl Sebacate	1	17	9.0×10^9
Dihexyl Sebacate	6	95	9.0×10^{11}
Di(2-Ethylhexyl)Sebacate	8	100	2.4×10^{12}
* The amount of hydrogen generated for n-hexadecane is set to 100.			

[0022] The present inventors have also found that a specific aromatic compound can effectively suppress hydrogen generation even when the compound has a specific volume resistivity exceeding $1.0 \times 10^{10} \Omega \cdot \text{cm}$.

[0023] Therefore, the compound of the present invention is at least one selected from the group consisting of (A) a compound having a specific volume resistivity of $1.0 \times 10^{10} \Omega \cdot \text{cm}$ or less, wherein the compound (A) is a diester selected

from the group consisting of dimethyl phthalate, dimethyl maleate, diethyl malonate, dibutyl malonate, and dihexyl malonate.

(Compound (A))

[0024] The compound (A) preferably has a specific volume resistivity of $5.0 \times 10^9 \Omega \cdot \text{cm}$ or less.

[0025] In addition, the compound (A) is preferably liquid at 25°C .

[0026] In addition, the compound (A) preferably has a dielectric constant ϵ of 3.0 or more at 500 MHz (25°C) and 1 GHz (25°C).

[0027] In addition, the compound (A) preferably has a Hansen solubility parameter polar term δ_p of 3.5 or more.

[0028] The term δ_p is expressed by the following formula, and δ_p increases as the dielectric constant ϵ increases. Generally, it is said that the dielectric constant ϵ of oil affects electron wave absorption, and it is said that the larger the dielectric constant ϵ and the larger the dielectric loss tangent, the more effectively electron waves can be absorbed, which can be a countermeasure against electron wave noise.

$$\delta_p^2 = \frac{12108}{V^2} \frac{\epsilon - 1}{2\epsilon + n_D^2} (n_D^2 + 2) \mu^2$$

[0029] It has been found that a compound having a specific volume resistivity of $1.0 \times 10^{10} \Omega \cdot \text{cm}$ has a δ_p of 3.5 or more. Therefore, it is considered that, when δ_p is 3.5 or more, hydrogen generation can be prevented and white band flaking can be prevented. The term δ_p is preferably 4.0 or more. The Hansen solubility parameter polar term δ_p is preferably 20 or less.

[0030] The term δ_p is preferably 3.5 or more, and the reason is as follows. Such a value makes it possible to achieve a conductivity to an extent sufficient to prevent charging and a high dielectric constant, and it is therefore considered that white band flaking can be suppressed through suppression of triboplasma generation.

[0031] The flash point of the compound of the present invention is preferably 70°C or lower because there is a risk of ignition by plasma generated due to friction of the lubrication portion. The flash point can be measured based on JIS K2265.

[Lubricant Composition]

[0032] Since being liquid at room temperature, the compound can be used alone as a lubricant composition, can also be used as a lubricant or a base oil of a grease, or can be mixed with a conventional base oil as a lubricant or a base oil of a grease to form a lubricant composition.

(Conventional Base Oil)

[0033] As the conventional base oil, one having a specific volume resistivity exceeding $1.0 \times 10^{10} \Omega \cdot \text{cm}$ can be used. One containing a saturated or unsaturated hydrocarbon group having 12 or more carbon atoms in total is preferable, and specific examples thereof include mineral oils and synthetic oils. As the mineral oil, it is possible to use a paraffinic mineral oil, a naphthenic mineral oil, or a mixture thereof. It is preferable to contain a highly refined mineral oil (that is, a mineral oil which has been subjected to dewaxing treatment to reduce wax component precipitation at low temperature, thereby lowering its pour point as compared with the pour point of unrefined mineral oils (-5°C to -20°C , measured according to JIS K 2269)). Examples of synthetic oils include synthetic hydrocarbons, ester oils, ether oils, glycol oils, silicone oils, and fluorinated oils. Examples of synthetic hydrocarbon oils include poly alpha olefins ("PAOs") and polybutene. Among these, poly alpha olefins are preferable. Examples of ester oils include diesters, trimellitate esters, and polyol esters. Examples of ether oils include alkyl diphenyl ethers ("ADEs"), dialkyl diphenyl ethers, and polypropylene glycol. Examples of glycol oils include polypropylene glycol and polypropylene alkyl ethers.

[0034] In the case of use in combination with a mineral oil or synthetic hydrocarbon (especially poly alpha olefin), hydrogen generation can be effectively suppressed even when the compound is in a small amount, for example more than 0.1% by mass, preferably more than 1% by mass, more preferably 2% by mass or more, and further preferably 3% by mass or more based on the total mass of the lubricant composition. The content of the compound in the lubricant composition of the present invention can be, for example, 40% by mass or less, 20% by mass or less, 10% by mass or less, 5% by mass or less, or 3% by mass or less. Considering the compatibility with the compounds described above, preferable conventional oils are ester oils such as diesters and polyol esters, ether oils such as alkyl phenyl ether oils, glycol oils such as water-insoluble polyalkylene glycols, silicone oils, fluorinated oils, and the like. From the viewpoints of resin resistance and heat resistance, mineral oils, synthetic oils, hydrocarbon oils, phenyl ether oils, and alkyl phenyl ether oils are preferable.

[0035] The kinematic viscosity at 40°C of the base oil in the lubricant composition of the present invention (that is, the compound (A) and/or (B) alone, or a mixture oil with the conventional oil) is preferably 10 to $500 \text{ mm}^2/\text{s}$. When the kinematic

viscosity at 40°C of the base oil is less than 10 mm²/s, it may be impossible to achieve a sufficient oil film at low speed or high temperature. Meanwhile, when the kinematic viscosity at 40°C of the base oil exceeds 500 mm²/s, there is a risk that the torque may rise at high speed or low temperature. For the same reasons, the range is more preferably 50 to 200 mm²/s and further preferably 60 to 130 mm²/s. Note that the kinematic viscosity of the base oil can be measured based on JIS K2283.

[0036] The content of the base oil in the lubricant composition of the present invention is preferably 60 to 99.9 parts by mass, more preferably 90 to 99.9 parts by mass, and further preferably 97 to 99.9 parts by mass relative to 100 parts by mass in total of the base oil and the anti-flaking agent. The content of the base oil is preferably in such ranges because of excellence in lubricity and low volatility.

(Optional Additive)

[0037] The lubricant composition of the present invention may further contain a general-purpose additive as necessary. For example, a rust inhibitor, a load-bearing additive, an antioxidant, and the like can be contained as necessary. The content of these optional additives is usually 0.5 to 5% by mass based on the total mass of the lubricant composition of the present invention.

[0038] Examples of the rust inhibitor include inorganic rust inhibitors and organic rust inhibitors. Examples of the inorganic rust inhibitors include inorganic metal salts such as sodium silicate, lithium carbonate, potassium carbonate, and zinc oxide. Examples of the organic rust inhibitors include benzoates such as sodium benzoate and lithium benzoate, sulfonates such as calcium sulfonate and zinc sulfonate, carboxylates such as zinc naphthenate and sodium sebacate, succinic acid derivatives such as succinic acid, succinic anhydride, and succinic acid half ester, sorbitan esters such as sorbitan monooleate and sorbitan trioleate, and fatty acid amine salts.

[0039] Examples of the load-bearing additive include phosphorus-containing ones such as phosphate esters, sulfur-based ones such as polysulfide and sulfurized oils and/or fats, phosphorus-sulfur-based ones such as phosphorothioates, thiocarbamates, thiophosphates, and organic phosphate esters.

[0040] The antioxidant is known to suppress oxidative degradation of grease, and examples thereof include phenol-based antioxidants and amine-based antioxidants.

[0041] Examples of the phenol-based antioxidants include 2,6-di-*tert*-butyl-*p*-cresol (BHT), 2,2'-methylenebis(4-methyl-6-*tert*-butylphenol), 4,4'-butylidenebis(3-methyl-6-*tert*-butylphenol), 2,6-di-*tert*-butyl-phenol, 2,4-dimethyl-6-*tert*-butylphenol, *tert*-butylhydroxyanisole (BHA), 4,4'-butylidenebis(3-methyl-6-*tert*-butylphenol), 4,4'-methylenebis(2,3-di-*tert*-butylphenol), 4,4'-thiobis(3-methyl-6-*tert*-butylphenol), and octadecyl-3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate. Among these, octadecyl-3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate is preferable.

[0042] Examples of the amine-based antioxidants include *N*-*n*-butyl-*p*-aminophenol, 4,4'-tetramethyl-di-aminodiphenylmethane, α -naphthylamine, *N*-phenyl- α -naphthylamine, phenothiazine, and alkyl diphenylamines. Among these, alkyl diphenylamines are preferable.

[0043] The lubricant composition of the present invention can be used as lubricating oil, conductive oil, dynamic pressure oil, and the like. The lubricant composition of the present invention is effective in preventing flaking wear.

[Grease Composition]

[0044] The lubricant composition of the present invention may further contain a thickener to form a grease composition.

[0045] For the same reasons as described for the lubricant composition, the content of the compound (A) and/or (B) is preferably more than 0.1% by mass, more preferably more than 1% by mass, further preferably 2% by mass or more, and particularly preferably 3% by mass or more based on the total mass of the grease composition of the present invention, and the upper limit can be, for example, 40% by mass or less, 20% by mass or less, 10% by mass or less, 5% by mass or less, or 3% by mass or less.

[0046] Examples of the thickener which can be used in the grease composition of the present invention include urea-based thickeners typified by diurea, lithium soap-based thickeners typified by lithium soap and lithium complex soap, and solid thickeners such as bentonite and silica gel. Urea-based thickeners and lithium soap-based thickeners are preferable.

[0047] The grease composition of the present invention may further contain a general-purpose additive as necessary. Examples of additives which can be used include ones described for the lubricant composition. The content of the optional additive is usually 0.1 to 5% by mass based on the total mass of the grease composition of the present invention.

(Penetration)

[0048] The worked penetration of the grease composition of the present invention is preferably 200 to 300 and more preferably 220 to 280. When the worked penetration exceeds 300, leakage due to high-speed rotation increases, which may result in failure to satisfy a sufficient lubrication life. Meanwhile, when the worked penetration is less than 200, the

fluidity of the grease is deteriorated, which may result in failure to satisfy a sufficient lubrication life. Note that, in the present specification, the term "penetration" refers to a 60-stroke worked penetration. The penetration can be measured according to JIS K2220-7.

5 (Content of Thickener)

[0049] The content of the thickener is preferably 5 to 25% by mass and more preferably 10 to 20% by mass based on the total mass of the grease composition of the present invention. When the content is less than 5% by mass, the grease is soft and may leak, which could result in failure to satisfy a sufficient lubrication life. Meanwhile, when the content is more than 10 25% by mass, the fluidity is inferior and thus it becomes difficult for the grease to enter the lubrication portion, which could result in failure to satisfy a sufficient lubrication life.

(Content of Base Oil)

15 **[0050]** The content of the base oil is preferably 60 to 90% by mass and more preferably 70 to 90% by mass based on the total mass of the grease composition of the present invention. The content of the base oil is preferably in such ranges because of excellence in lubricity and low volatility.

[Bearing]

20 **[0051]** The grease composition of the present invention is used in various rolling bearings for industrial machines and automobiles. Examples for industrial machines include rolling bearings in various motors for industrial machines, reducers and hydraulic equipment of industrial robots, main shafts and reducers of wind power generators, and peripherals of elevator hoists. The use for automobiles is preferably a rolling bearing for automobile electrical equipment and auxiliaries. 25 Examples of the automobile electrical equipment and auxiliaries include alternators, electromagnetic clutches for automobile air conditioners, intermediate pulleys, idler pulleys, and tension pulleys.

[Examples]

30 [Hydrogen Generation Test and Measurement of Amount of Hydrogen Generated]

[0052] The amount of hydrogen generated was measured according to the method described in Nouyama, Nakayama, et al., Manuscript Preparation for Tribology Conference, Tokyo (2017), 185.

[0053] Specifically, a triboplasma generator (Fig. 1) was used capable of generating triboplasma between the needle and the flat plate electrode. The needle was the cathode and the flat plate was the anode. The material of the needle was 35 SCM435 steel and the apex angle of the needle was 120°. The needle was arranged perpendicular to the anode flat plate, and was fixed at a position where the distance between the tip of the needle and the upper surface of the anode was 50 μm. The distance between the needle and the flat plate electrode was controlled by a micrometer. The material of the anode flat plate was SPCC steel. The anode flat plate constituted the bottom portion inside the container. The container was charged 40 with the anti-flaking agent and the like of Examples or Comparative Examples, and the needle was in contact with the anti-flaking agent and the like inside the container. The anode flat plate and the cathode needle were connected by a high voltage power source. The voltage and current when a voltage was applied was measurable by an oscilloscope. The container and the needle were surrounded by a larger casing (hereinafter referred to as the "atmosphere control chamber") so as to cover both. The top portion of the atmosphere control chamber had an opening provided therein, and the gas inside 45 the atmosphere control chamber was collectable through a microsyringe. The upper side portion of the atmosphere control chamber also had an opening provided therein so as to introduce dry air therethrough. The gas inside the atmosphere control chamber was detectable by a semiconductor sensor.

[0054] Dry air was introduced for 30 seconds to replace the gas inside the atmosphere control chamber. After the gas inside the atmosphere control chamber was replaced with dry air, the atmosphere control chamber was subjected to 50 discharging for 30 seconds while monitoring the current value and the voltage value with an oscilloscope, and then left for 20 seconds to collect the generated gas through a microsyringe. The collected gas was introduced into gas chromatography to measure the amount of hydrogen gas. Note that the gas chromatography was measured using a gas chromatograph GC-2010 (manufactured by Shimadzu Corporation), a column RT-Msieve φ0.43 mm × 30 m, and a detector TCD. The amount of hydrogen generated for each compound was calculated with the amount of hydrogen 55 generated for n-hexadecane set to 100%.

[0055] Tables 3 to 10 present the results. Examples 1 to 38 are examples of the anti-flaking agent, and Examples 39 to 71 are examples of the lubricating oil composition containing the anti-flaking agent. Example 42 is a mixture of 3.0% by mass of dimethyl malonate of Example 3 and 97.0% by mass of poly alpha olefin of Comparative Example 8, and indicates that,

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even when the specific volume resistivity of the mixture exceeds $1.0 \times 10^{10} \Omega \cdot \text{cm}$, the amount of hydrogen generated can be suppressed to 0% if a predetermined amount of the anti-flaking agent of the present application having a specific volume resistivity of $1.0 \times 10^{10} \Omega \cdot \text{cm}$ or less is contained.

[0056] Examples 1 to 3, 6 to 11, 15 to 39 and 41 to 71 are comparative.

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Table 3

		Ex.	Ex.	Ex.	Ex.	Ex.	Ref. Ex.	Ref. Ex.	Ref. Ex.	Ref. Ex.	Ref. Ex.	
		1	2	3	4	5	6	7	8	9	10	
10	Aromatic Hydrocarbon Compound	Diphenylmethane	100									
		Diphenylpropane		100								
		Cis-1,2-Diphenylethylene			100							
15	Monoester	Methyl Myristate										
20	Diester	Dimethyl Phthalate				100						
		Dimethyl Maleate					100					
		Dimethyl Malonate						100				
		Dimethyl Succinate							100			
		25	Dimethyl Glutarate							100		
		Dimethyl Adipate									100	
		Dimethyl Sebacate										100
		30	Diethyl Malonate									
		Dibutyl Malonate										
		Dihexyl Malonate										
		35	Dihexyl Sebacate									
		Di-2-Ethylhexyl Sebacate										
40	Triester	Tributyl Trimellitate										
Tetraester	Pentaerythritol											
45	Glycol	Tetraethylene Glycol										
		Tripropylene Glycol										
		Tetraethylene Glycol Dimethyl Ether										
		Poly (Oxyethylene) Glycol										
		50	Polypropylene Glycol Monobutyl Ether									
		Poly(Oxypropylene, Oxybutylene)Glycol										
55	S-cont. cmd.	Dibutyl Sulfoxide										
		2,2'-Thiodiethanol										

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(continued)

Dielectric Constant 500 MHz	3.2	-	-	8.8	10.2	11.2	8.5	9.1	7.9	-
Dielectric Constant 1 GHz	3.3	-	-	8.0	10.1	11.3	8.5	9.1	8.0	-
Hansen Parameter Polar Force δP	1.0	2.1	1.9	7.8	10.4	7.0	6.6	6.4	5.6	5.1
Amount of Hydrogen Generated %	2	3	2	0	0	0	0	0	0	0

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Table 4

		Ref. Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.
		11	12	13	14	15	16	17	18	19	20
5											
	Aromatic Hydrocarbon Compound	Diphenylmethane									
		Diphenylpropane									
10		<i>Cis</i> -1,2-Diphenylethylene									
	Monoester	Methyl Myristate									
	Diester	Dimethyl Phthalate									
15		Dimethyl Maleate									
		Dimethyl Malonate									
		Dimethyl Succinate									
20		Dimethyl Glutarate									
		Dimethyl Adipate									
		Dimethyl Sebacate	100								
25		Diethyl Malonate		100							
		Dibutyl Malonate			100						
		Dihexyl Malonate				100					
30		Dihexyl Sebacate									
		Di-2-Ethylhexyl Sebacate									
	Triester	Tributyl Trimellitate									
	Tetraester	Pentaerythritol									
35	Glycol	Tetraethylene Glycol				100					
		Tripropylene Glycol					100				
		Tetraethylene Glycol Dimethyl Ether						100			
40		Poly (Oxyethylene) Glycol							100		
		Polypropylene Glycol Monobutyl Ether								100	
45		Poly(Oxypropylene, Oxybutylene)Glycol									
	S-cont. cmd.	Dibutyl Sulfoxide									100
50		2,2'-Thiodiethanol									

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(continued)

Dielectric Constant 500 MHz	6.4	9.0	-	-	15.6	7.8	9.0	18.1	4.6	47.6
Dielectric Constant 1 GHz	6.4	9.0	-	-	12.3	6.3	9.0	13.9	4.3	47.3
Hansen Parameter Polar Force δP	4.5	6.0	4.4	3.8	9.4	7.8	6.0	9.4	7.8	16.4
Amount of Hydrogen Generated %	17	0	0	1	0	0	0	0	6	0

Table 5

	Ex. 21	Ex. 22	Ex. 23	Ex. 24	Ex. 25	Ex. 26	Ex. 27	Ex. 28	Ex. 29	Ex. 30	
Aromatic Hydrocarbon Compound	Diphenylmethane										
	Diphenylpropane										
	Cis-1,2-Diphenylethylene										
Monoester	Methyl Myristate										
	Dimethyl Phthalate										
	Dimethyl Maleate										
	Dimethyl Malonate										
	Dimethyl Succinate										
	Dimethyl Glutarate										
	Dimethyl Adipate										
	Dimethyl Suberate										
	Dimethyl Sebacate										
	Diethyl Malonate										
Diester	Dibutyl Malonate										
	Dihexyl Malonate										
	Dihexyl Sebacate										
	Di-2-Ethylhexyl Sebacate										
	Tributyl Trimellitate										
	Pentaerythritol										
	Tetraester	Tetraethylene Glycol									
		Tripropylene Glycol									
	Glycol	Tetraethylene Glycol Dimethyl Ether									
		Poly(Oxyethylene)Glycol									
Polypropylene Glycol Monobutyl Ether											
Poly(Oxypropylene, Oxybutylene)Glycol											

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(continued)

	Ex. 21	Ex. 22	Ex. 23	Ex. 24	Ex. 25	Ex. 26	Ex. 27	Ex. 28	Ex. 29	Ex. 30
S-cont. cmd.										
	100									
P-cont. cmd.		100								
			100							
N-cont. cmd.			100							
				100						
					100					
						100				
							100			
							100			

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Table 6

		Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.
		31	32	33	34	35	36	37	38	39	40
5	Aromatic Hydrocarbon Compound	Diphenylmethane									
		Diphenylpropane									
		Cis-1,2-Diphenylethylene									
10	Monoester	Methyl Myristate									
	Diester	Dimethyl Phthalate								3.0	
		Dimethyl Maleate									3.0
15		Dimethyl Malonate									
		Dimethyl Succinate									
		Dimethyl Glutarate									
		Dimethyl Adipate									
20		Dimethyl Suberate									
		Dimethyl Sebacate									
		Diethyl Malonate									
25		Dibutyl Malonate									
		Dihexyl Malonate									
		Dihexyl Sebacate									
	Di-2-Ethylhexyl Sebacate										
30	Triester	Tributyl Trimellitate									
	Tetraester	Pentaerythritol									
35	Glycol	Tetraethylene Glycol									
		Tripropylene Glycol									
		Tetraethylene Glycol Dimethyl Ether									
		Poly(Oxyethylene)Glycol									
		Polypropylene Glycol Monobutyl Ether									
40		Poly(Oxypropylene, Oxybutylene)Glycol									
	S-cont. cmd.	Dibutyl Sulfoxide									
		2,2'-Thiodiethanol									
	P-cont. cmd.	Trimethyl Phosphate									

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Table 7

	Ex.41	Ex.42	Ex.43	Ex.44	Ex.45	Ex.46	Ex.47	Ex.48	Ex.49	Ex.50
Aromatic Hydrocarbon Compound	Diphenylmethane									
	Diphenylpropane									
	Cis-1,2-Diphenylethylene									
Monoester	Methyl Myristate									
	Dimethyl Phthalate									
	Dimethyl Maleate									
	Dimethyl Malonate	3.0	3.0	40.0						
	Dimethyl Succinate				5.0	10.0				
	Dimethyl Glutarate						5.0			
	Dimethyl Adipate									
	Dimethyl Suberate									
	Dimethyl Sebacate									
	Diethyl Malonate							10.0		
Diester	Dibutyl Malonate									
	Dihexyl Malonate									
	Dihexyl Sebacate									
	Di-2-Ethylhexyl Sebacate			60.0						
	Tributyl Trimellitate									
	Pentaerythritol									
	Tetraethylene Glycol							1.0		
	Tripropylene Glycol								10.0	
	Tetraethylene Glycol Dimethyl Ether									10.0
	Poly(Oxyethylene)Glycol									
Glycol	Polypropylene Glycol Monobutyl Ether									
	Poly(Oxypropylene, Oxybutylene)Glycol									

5	0
10	0
15	0
20	0
25	0
30	0
35	
40	Amount of Hydrogen Generated %
45	
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(continued)

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Table 8

		Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.
		51	52	53	54	55	56	57	58	59	60
5	Aromatic Hydrocarbon Compound	Diphenylmethane									
		Diphenylpropane									
		<i>Cis</i> -1,2-Diphenylethylene									
10	Monoester	Methyl Myristate									
		Dimethyl Phthalate									
		Dimethyl Maleate									
15		Dimethyl Malonate									
		Dimethyl Succinate									
		Dimethyl Glutarate									
		Dimethyl Adipate									
20	Diester	Dimethyl Sebacate									
		Dimethyl Suberate									
		Diethyl Malonate									
25		Dibutyl Malonate									
		Diethyl Sebacate									
		Dihexyl Malonate									
		Dihexyl Sebacate									
30		Di-2-Ethylhexyl Sebacate	99.0			97.0			90.0		
	Triester	Tributyl Trimellitate									
	Tetraester	Pentaerythritol									
35	Glycol	Tetraethylene Glycol									
		Tripropylene Glycol									
		Tetraethylene Glycol Dimethyl Ether									
40		Poly (Oxyethylene) Glycol	1.0	0.5	1.0						
		Polypropylene Glycol Monobutyl Ether									
		Poly(Oxypropylene, Oxybutylene)Glycol									
45	S-cont. cmd.	Dibutyl Sulfoxide				3.0	3.0	3.0			
		2,2'-Thiodiethanol							3.0		
50	P-cont. cmd.	Trimethyl Phosphate							10.0	10.0	10.0
	N-cont. cmd.	Formamide									
		<i>N</i> -Methylformamide									
55		<i>N-tert</i> -Butylformamide									
		Tetramethylurea									
		Tetraethylurea									

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(continued)

5	General Anti-static Agent	Poly(Oxyethylene)Alkylamine									
		Glycerin Aliphatic Ester Monocaprylin									
10	Ionic Liquid	(N-(Methoxyethyl)-1-Methylpyrrolidinium Bis(Tri-fluoromethylsulfonyl)Imide)									
		Liquid Crystal	4-Cyano-4'-Pentylbiphenyl								
	Mineral Oil	P-Based Mineral Oil									
15	Synthetic Hydrocarbon	PAO		99.5			97.0		97.0		90.0
		Ether	ADE			99.0			97.0		
Alkyl Tetraphenyl Ether											
Pentaphenyl Ether											
Tetraphenyl Ether											
20	SP-cont. cmd.	Alkylated Triphenyl Phosphorothioate									
25	NS-Based Compound	Dimercaptothiadiazole Derivative									
		MoDTC									
30	Fatty Acid Amine Salt	Oleic Acid Dicycloamine Salt									
		Number of Aromatic Carbon Atoms %	-	-	-	-	-	-	-	-	-
		Specific Volume Resistivity $\Omega \cdot \text{cm}$	-	-	-	-	-	-	-	-	-
		Dielectric Constant 500 MHz	-	-	-	-	-	-	-	-	-
35		Dielectric Constant 1 GHz	-	-	-	-	-	-	-	-	-
		Hansen Parameter Polar Force δP	-	-	-	-	-	-	-	-	-
		Amount of Hydrogen Generated %	0	0	0	0	0	0	0	0	0

Table 9

		Ex. 61	Ex. 62	Ex. 63	Ex. 64	Ex. 65	Ex. 66	Ex. 67	Ex. 68	Ex. 69	Ex. 70	Ex. 71	
45	Aromatic Hydrocarbon Compound	Diphenylmethane											
		Diphenylpropane											
		Cis-1,2-Diphenylethylene											
50	Monoester	Methyl Myristate											
55		Dimethyl Phthalate											
		Dimethyl Maleate											
		Dimethyl Malonate											
		Dimethyl Succinate											
		Dimethyl Glutarate											
		Dimethyl Adipate											

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(continued)

		Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.	Ex.
		61	62	63	64	65	66	67	68	69	70	71
5	Diester	Dimethyl Suberate										
		Dimethyl Sebacate										
		Diethyl Malonate										
10		Dibutyl Malonate										
		Dihexyl Malonate										
		Dihexyl Sebacate										
15		Di-2-Ethylhexyl Sebacate	99.0								99.0	
	Triester	Tributyl Trimellitate										
	Tetraester	Pentaerythritol										
20	Glycol	Tetraethylene Glycol										
		Tripropylene Glycol										
		Tetraethylene Glycol Dimethyl Ether										
25		Poly(Oxyethylene) Glycol										
		Polypropylene Glycol Monobutyl Ether										
30		Poly (Oxypropylene, Oxybutylene) Glycol										
35	S-cont. cmd.	Dibutyl Sulfoxide										
		2,2'-Thiodiethanol										

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5	0
10	0
15	0
20	0
25	0
30	0
35	
40	Amount of Hydrogen Generated %
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(continued)

Table 10

	Comp. Ex. 1	Comp. Ex.2	Comp. Ex.3	Comp. Ex.4	Comp. Ex.5	Comp. Ex.6	Comp. Ex.7	Comp. Ex.8	Comp. Ex.9	Comp. Ex. 10
Aromatic Hydrocarbon Compound	Diphenylmethane									
	Diphenylpropane									
	Cis-1,2-Diphenylethylene									
Monoester	Methyl Myristate	100								
	Dimethyl Phthalate									
Diester	Dimethyl Maleate									
	Dimethyl Malonate									
	Dimethyl Succinate									
	Dimethyl Glutarate									
	Dimethyl Adipate									
	Dimethyl Suberate									
	Dimethyl Sebacate									
	Diethyl Malonate									
	Dibutyl Malonate									
	Dihexyl Malonate		100							
Triester	Dihexyl Sebacate									
	Di-2-Ethylhexyl Sebacate		100							
Tetraester	Tributyl Trimellitate			100						
	Pentaerythritol								100	

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(continued)

Comp. Ex. 1	Comp. Ex.2	Comp. Ex.3	Comp. Ex.4	Comp. Ex.5	Comp. Ex.6	Comp. Ex.7	Comp. Ex.8	Comp. Ex.9	Comp. Ex. 10
Tetraethylene Glycol									
Tripropylene Glycol									
Tetraethylene Glycol Dimethyl Ether									
Poly(Oxyethylene)Glycol									
Polypropylene Glycol Monobutyl Ether									
Poly(Oxypropylene, Oxybutylene)Glycol					100				
Dibutyl Sulfoxide									
2,2'-Thiodiethanol									
Trimethyl Phosphate									
S-cont. cmd.									
P-cont. cmd.									

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(continued)

Dielectric Constant 1 GHz	4.1	-	3.9	-	3.5	3.5	2.8	2.8	2.9	-
Hansen Parameter Polar Force δP	2.7	3.0	2.1	6.8	2.1	8.1	1.0	1.0	25.0	-
Amount of Hydrogen Generated %	105	95	100	77	88	101	100	106	93	100

[0057] The suppliers and trade names of the compounds used in Examples and Comparative Examples are presented below.

5	Tetraester	: Ester of pentaerythritol with carboxylic acid (manufactured by Kao Corporation under the trade name of "KAOLUBE 279")
	Glycol	: Poly(oxyethylene)glycol (manufactured by NOF Corporation under the trade name of "PEG #200")
10		: Polypropylene glycol monobutyl ether (manufactured by NOF Corporation under the trade name of "UNILUBE MB-19")
		: Poly(oxypropylene, oxybutylene)glycol monodecyl ether (manufactured by Dow Chemical Company under the trade name of "OSP-68")
	Antistatic Agent	: Poly(oxyethylene)laurylamine (manufactured by Nippon Nyukazai Co., Ltd. under the trade name of "Newcol LA-407")
15		: Glycerin aliphatic ester monocaprylin (manufactured by Riken Vitamin Co., Ltd. under the trade name of "Poem M-100")
	Ionic Liquid	: <i>N</i> -(Methoxyethyl)-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide (manufactured by Merck)
20	Liquid Crystal	: 4-Cyano-4'-pentylbiphenyl (manufactured by Tokyo Chemical Industry Co., Ltd. under the trade name of "5CB")
	Mineral Oil	: P-based mineral oil (manufactured by JX Energy under the trade name of "Super Oil K-100")
	Synthetic Hydrocarbon Ether Oil	: PAO8 (kinematic viscosity at 40°C is 45 mm ² /s)
25		: ADE (manufactured by MORESCO under the trade name of "LB-100")
		: Alkyl tetraphenyl ether (manufactured by MORESCO under the trade name of "s-3101")
		: Pentaphenyl ether (manufactured by MORESCO under the trade name of "s-3105")
		: Tetraphenyl ether (manufactured by MORESCO under the trade name of "s-3103")
30	SP-Based Compound	: Alkylated triphenyl phosphorothionate (manufactured by BASF Japan under the trade name of "IRGALUBE 211")
	NS-Based Compound	: Dimercaptiothiadiazole derivative (manufactured by The Elco Corporation under the trade name of "Elco 461")
		: MoDTC (manufactured by ADEKA Corporation under the trade name of "SAKURALUBE 525")
35	Fatty Acid Amine Salt	: Oleic acid dicycloamine salt (manufactured by Albess Co., Ltd. under the trade name of "NONRUST Z-1000")

40 Claims

1. Use as an anti-flaking agent of at least one selected from the group consisting of (A) a compound having a specific volume resistivity of $1.0 \times 10^{10} \Omega \cdot \text{cm}$ or less, wherein the compound (A) is a diester selected from the group consisting of dimethyl phthalate, dimethyl maleate, diethyl malonate, dibutyl malonate, and dihexyl malonate.
2. Lubricant composition comprising a diester selected from the group consisting of dimethyl maleate, dibutyl malonate, and dihexyl malonate and at least one conventional base oil selected from the group consisting of mineral oils and synthetic oils.
3. The lubricant composition according to claim 2, wherein the base oil is at least one selected from the group consisting of mineral oils, synthetic hydrocarbon oils, and ether oils.
4. The lubricant composition according to any one of claims 2 to 3, wherein a content of the compound exceeds 0.1% by mass based on a total mass of the composition.
5. The lubricant composition according to any one of claims 2 to 4, further comprising a thickener.

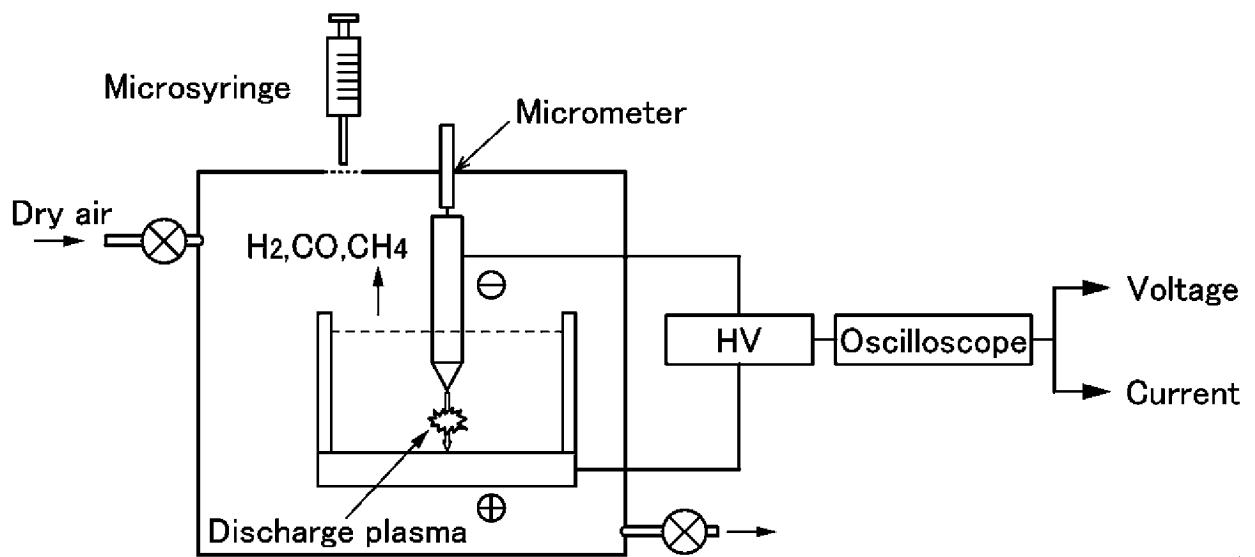
Patentansprüche

- 5 1. Verwendung als Antischuppenmittel von mindestens einem aus der Gruppe bestehend aus (A) eine Verbindung mit einem spezifischen Volumenwiderstand von $1,0 \times 10^{10} \Omega \cdot \text{cm}$ oder weniger, wobei die Verbindung (A) ein Diester ist, ausgewählt aus der Gruppe, bestehend aus Dimethylphthalat, Maleinsäuredimethylester, Malonsäurediethylester, Malonsäuredibutylester und Malonsäuredihexylester.
- 10 2. Schmiermittelzusammensetzung, umfassend einen Diester, ausgewählt aus der Gruppe, bestehend aus Maleinsäuredimethylester, Malonsäuredibutylester und Malonsäuredihexylester, und mindestens ein konventionelles Grundöl, ausgewählt aus der Gruppe, bestehend aus Mineralölen und synthetischen Ölen.
- 15 3. Schmierstoffzusammensetzung nach Anspruch 2, wobei das Grundöl mindestens eines ist, ausgewählt aus der Gruppe, bestehend aus Mineralölen, synthetischen Kohlenwasserstoffölen und Etherölen.
- 20 4. Schmiermittelzusammensetzung nach einem der Ansprüche 2 bis 3, wobei ein Gehalt der Verbindung 0,1 Masse-%, bezogen auf eine Gesamtmasse der Zusammensetzung, übersteigt.
5. Schmiermittelzusammensetzung nach einem der Ansprüche 2 bis 4, ferner umfassend ein Verdickungsmittel.

Revendications

- 25 1. Utilisation comme agent anti-écaillage d'au moins un élément choisi dans le groupe constitué (A) d'un composé ayant une résistivité volumique spécifique de $1,0 \times 10^{10} \Omega \text{ cm}$ ou moins, dans laquelle le composé (A) est un diester choisi dans le groupe constitué du phtalate de diméthyle, du maléate de diméthyle, du malonate de diéthyle, du malonate de dibutyle, et du malonate de dihexyle.
- 30 2. Composition de lubrifiant comprenant un diester choisi dans le groupe composé du maléate de diméthyle, du malonate de dibutyle et du malonate de dihexyle et au moins une huile de base classique choisie dans un groupe composé d'huiles minérales et d'huiles synthétiques.
- 35 3. Composition de lubrifiant selon la revendication 2, dans laquelle l'huile de base est au moins une huile choisie dans le groupe constitué d'huiles minérales, d'huiles hydrocarbonées synthétiques et d'huiles éther.
- 40 4. Composition de lubrifiant selon l'une quelconque des revendications 2 et 3, dans laquelle la teneur en composé dépasse 0,1 % en masse sur la base d'une masse totale de la composition.
- 45 5. Composition de lubrifiant selon l'une quelconque des revendications 2 à 4, comprenant également un épaississant.
- 50
- 55

FIG.1



REFERENCES CITED IN THE DESCRIPTION

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