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(54) Titre : COMPOSITION DE LUBRIFIANT COMPRENANT DU SILASESQUIOXANE
(54) Title: LUBRICANT COMPOSITION COMPRISING SILASESQUIOXANE

(57) Abrégé/Abstract:
The invention relates to a lubricant composition for application onto the surface of drive elements, the lubricant composition containing a base oil and a silasesquioxane. The composition is suitable for preventing, reducing or avoiding fatigue phenomena in the material of drive elements, such as gray staining, false brinelling and white etching cracks.

Abstract

The invention relates to a lubricant composition for application onto the surface of drive elements, the lubricant composition containing a base oil and a silasesquioxane. The composition is suitable for preventing, reducing or avoiding fatigue phenomena in the material of drive elements, such as gray staining, false brinelling and white etching cracks.

Lubricant Composition Comprising Silasesquioxane

Field

The present disclosure generally relates to a lubricant composition for application onto a surface of drive elements, such as rolling bearings, transmissions, friction bearings, and chains. The composition may be suitable for preventing, reducing, or avoiding fatigue phenomena in the material of drive elements, such as gray staining, false brinelling, and white etching cracks. The present disclosure also generally relates to a use of the lubricant composition for treating surfaces of drive elements as well as the further use of such drive elements.

Background

Two types of damage occur in drive elements in the case of excessive mechanical stress:

1) Etching and wear, where the damage starts at the surface of the contact areas.

2) Fatigue damage, which starts in the structure below the stressed areas and ultimately results in blowouts such as gray staining, false brinelling, and white etching cracks.

To reduce or avoid wear and etching, there are a number of additives and solid lubricants which are well known and used frequently.

Only a few effective measures are known to prevent damage from fatigue. One measure is to increase the thickness of the lubrication film.

Wear due to fatigue is created by the local overstress of the material by periodic compressive stress. The fatigue of the material becomes visible in the form of

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gray spots (gray staining, surface fatigue, micro-pitting) or grooves on the surface of the material. Generally, fine cracks occur in the metal lattice, initially 20 to 40 µm below the surface, which lead to material blowouts. The small, microscopically visible blowouts on the tooth flank referred to as micro-pitting or gray staining appear macroscopically as matte gray areas. In gearings, gray staining can be observed at almost all speed ranges. In rolling gears as well, very flat blowouts appear on the track in the area of the sliding contact as gray staining as well. These correlations are described in detail in DE 10 2007 036 856 A1 and the literature cited there.

White etching cracks (WEC) may result in fatigue damage that occurs much sooner than expected in a drive element with particular stress parameters. Metallographically, white cracks can be detected in the depths of the structure. The white staining is due to the fact that the cracks that appear as white were not subjected to the etching that is required in the sample preparation. Under continued tribological load, these cracks may lead to blowouts in the material and the failure of the components. Several factors such as slip, harmful currents, and diffused hydrogen have been discussed as the cause.

It is known that such damage occurs especially in the gearing mechanisms of electric motors or generators of wind turbines (also refer to the dissertation by H. Surborg, 2014, Otto von Guericke University Magdeburg, Shaker Verlag Aachen 2014).

False brinelling is a type of damage that occurs in seemingly non-moving bearings. Due to vibrations (for example in machines but also during the transport by motor vehicles, rail vehicles, or ships) or elastic deformations, the micromovements are transmitted to the contact areas which may cause damage

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after just a few load changes. This may result in an uneven operating behavior and the immediate or early failure of the component.

In general, the damaging mechanisms described above that are caused by the cracks are some of the most serious material impairments.

To avoid such fatigue damage, the following steps are usually taken in practice:

- Decrease of the contact forces,
- Suitable selection of the lubricant,
- Sufficient lubricant supply,
- Favorable positioning and design of the lubrication points,
- Avoidance of states without lubrication.

Furthermore, different additives are used to improve the viscosity properties in lubricants to avoid or at least minimize the aforementioned damage in rolling gears, gearwheels, transmissions, and the like.

In addition, various tests have been performed to avoid fatigue phenomena. The attempt was made, for example, to improve the lubricating effect of lubricants by adding various additives. The tests included in particular additives with which the friction between components can be reduced or that have an improved viscosity.

DE-OS 1 644 934, for example, describes organophosphates as additives in lubricants, which are added as anti-fatigue additives.

The aforementioned DE 10 2007 036 856 A1 discloses the addition of polymers with ester groups that are used as anti-fatigue additives in lubricants.

US 2003/0092585 A1 discloses thiazoles as additives that can prevent surfaces from being damaged.

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EP 1 642 957 A1 relates to the use of MoS2 and molybdenum dithiocarbamate, which are used as additives in urea greases for drive shafts.

The additives described above such as organophosphates and thiazoles are organic substances and therefore not thermally stable. Furthermore, they may evaporate under operating conditions or, as traditional anti-wear additives, react especially with the metal surfaces, i.e., they react primarily on the roughness peaks that are touching because, due to the flash temperatures that occur there, sufficient energy is present for a chemical reaction with the metallic friction layer. At the most, therefore, they can counteract fatigue damage only as a secondary function. Solid lubricants such as molybdenum disulfide, however, tend to sediment from oil preparations due to their density and may also have a corrosive effect. Since solid particles with particle sizes in the micrometer range are used, the flow properties are significantly influenced and the viscosity increases. Newton's flow properties are deviated from as well. This decreases the availability of the additive in the lubrication gap. REM testing on the surface of the metal friction partners have shown that these structures have recesses with dimensions of significantly below 1 µm. These recesses are not accessible to the solid lubricant particles with sizes in the micrometer range.

DE 102011103215 A1 describes the use of a composition containing surface-modifying nanoparticles and a carrier material that is applied to the surfaces of drive elements to prevent or reduce fatigue damage. It is assumed that the mode of action of the nanoparticles is based on the fact that they attach to the surface of the drive elements thereby making it smoother. The smoothening increases the contact surfaces and reduces the surface pressure.

JP 2006144827 A refers to compositions with silica nanoparticles to suppress SEC damage.

What is disadvantageous about the compositions described in DE 102011103215 A1 and JP 2006144827 A is that the methods that are available often make it difficult to cause a sufficient configuration of the OH groups on the surface of the nanoparticles. This may cause stability problems during storage. Furthermore, the inclusion of air may cause the formation of foam. Finally, filtration problems may arise when the lubricant is filtered.

The above information is presented as background information only to assist with an understanding of the present disclosure. No assertion or admission is made as to whether any of the above, or anything else in the present disclosure, unless explicitly stated, might be applicable as prior art with regard to the present disclosure.

Summary

According to an aspect of the present disclosure, a lubricant composition for application onto a surface of drive elements is provided. The lubricant composition may comprise a base oil, and a silasesquioxane. In an embodiment, the silasesquioxane has chemical formula [RSiO3/2]n, wherein n = 6, 8, 10, or 12, and/or R comprises an oxirane polymer with a degree of polymerization of 4 to 20 repetition units.

The foregoing summary provides some aspects and features according to the present disclosure but is not intended to be limiting. Other aspects and features of the present disclosure will become apparent to those ordinarily skilled in the art upon review of the following description of specific embodiments.

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Accordingly, the detailed description is to be regarded as illustrative in nature and not restrictive.

Detailed Description

In an aspect, the present disclosure relates to a lubricant composition that can be applied onto the surfaces of drive elements to prevent, reduce, or avoid fatigue phenomena such as gray staining, false brinelling, and white etching cracks and that at least partially eliminates the disadvantages that occur in prior art.

The present disclosure relates to a lubricant composition for application onto the surface of drive elements, the lubricant composition containing a base oil and a silasesquioxane. The composition is suitable for preventing, reducing, or avoiding fatigue phenomena in the material of drive elements such as gray staining, false brinelling, and white etching cracks. The positive influence of the lubricant composition is surprising because silasesquioxane is significantly smaller than the nanoparticles described in the printed documents DE 102011103215 A1 and JP2006144827A, and it was therefore not possible to assume that they can smoothen a surface in the same way as these particles and therefore reduce the fatigue phenomena.

It was found in field tests that the composition according to the present disclosure can be homogeneously mixed with base oils of all types of polarities since, for example by selecting the substituents of the silasesquioxane, the polarity of the composition is easy to adapt. In addition, it is possible to guarantee a sufficient saturation of the OH groups of the silasesquioxane due to its production process. It was also found that the use of silasesquioxane results in a high storage stability and that the foaming behavior or the filtration capability are not impaired. It was also found in field tests that silasesquioxanes that are liquid at room temperature (20°C) and/or have low melting points (preferably below 100°C, DIN EN ISO 11357-2 (edition: 2014-07)) can be used in a favorable manner.

Silasesquioxane are organosilicon compounds and form cage-like structures with Si-O-Si bonds and tetrahedral Si-corners. The silasesquioxane may have 6 to 12 Si-corners in its molecular form and/or be present as an oligomer and/or polymer. What is preferred according to the present disclosure are molecular silasesquioxanes; even more preferred are molecular silasesquioxanes with 6 to 12 Si-corners, even more preferred 7 to 10 and especially 7 to 8 Si-corners. In a preferred embodiment, every Si-center is bonded with three oxo groups, which, in turn, bond with other Si-centers.

In another preferred embodiment, the Si-centers are only partially bonded with three oxo groups, which are bonded with other Si-centers, and preferably 3 si-centers are bonded with only two oxo groups that are bonded with other Si-centers. The third group is preferably a substituent here, and even more preferably a hydroxy substituent.

The fourth group on the Si is also preferably a substituent, which makes it possible to obtain a surface-modified silasesquioxane, which is preferred according to the present disclosure. Suitable substituents are, for example, alkyl (C1-C20), cycloalkyl (C3-C20), alkenyl (C2-C20), cycloalkenyl (C5-C20), alkinyl (C2-C20), cycloalkinyl (C5-C20), aryl (C6-C18), or the heteroaryl group, oxy, hydroxy, alkoxy (C4-C10), oxirane polymer (degree of polymerization with 4 to 20 repetition units), carboxy, silyl, alkyl silyl, alcoxy silyl, siloxy, alkyl siloxy, alkoxy siloxy, silyl alkyl, alkoxy silyl alkyl, alkyl silyl alkyl, halogen, epoxy (C2-C20), ester, aryl ether, fluoroalkyl, blocked isocyanate, acrylate, methacrylate, mercapto, nitrile, amine, and/or phosphine group, each substituted or unsubstituted. Here, the silasesquioxane may comprise same substituents or mixes of different substituents.

Preferred substituents are hydroxy, alkyl (C4-C10), aryl (C6-C12), especially phenyl and tolyl, alkoxyl (C4-C10), alkenyl (C2-C10), oxirane polymer, especially polyethylene glycol, polypropylene glycol, polybutylene glycol, and/or their copolymers (degree of polymerization 4 to 20, especially 10 to 15 repetition units), epoxy (C2-C10), and/or cycloalkyl (C5-C10), each substituted or unsubstituted.

Especially preferred substituents are hydroxy, alkyl (C4-C10), phenyl, tolyl, alkoxyl (C4-C10), alkenyl (C2-C10), and/or oxirane polymer, especially polyethylene glycol, polypropylene glycol and/or their copolymers (degree of polymerization 4 to 20, especially 10 to 15 repetition units), each substituted or unsubstituted.

In another embodiment of the present disclosure, R may comprise additional functional groups, especially thiol groups, phosphate groups individually or in combination. The optionally present thiol or phosphate groups may additionally react with the metal surface to be protected.

According to the present disclosure, the silasesquioxane may also be mixtures of structurally different silasesquioxanes.

Silasesquioxane may, for example, be synthesized by hydrolysis of organic trichlorosilane (idealized: $8\ RSiCl_3; + 12\ H_2O \rightarrow [RSiO_{3/2}]_8 + 24\ HCl$). Depending on the substituent (R), the exterior of the cage may be modified further. If R = H, the Si-H group may be subjected to a hydrolyzation or oxidation to silanol. The easiest way to make bridged poly-silasesquioxane is to use clusters containing two or more tri-functional silyl groups that are not bonded with hydrolyzable silicon carbon bonds. Vinyl-substituted silasesquioxane can be bound by the alkene metathesis.

In a preferred embodiment, the silasesquioxane has the chemical formula $[RSiO_{3/2}]_n$ with: n = 6, 8, 10, 12; preferably n = 8, 10, 12 and especially 8, with R being independently from each other = alkyl (C1-C20), cycloalkyl (C3-C20), alkenyl (C2-C20), cycloalkenyl (C5-C20), alkinyl (C2-C20), cycloalkinyl (C5-C20), aryl (C6-C18), or the heteroaryl group, oxy, hydroxy, alkoxy (C4-C10), oxirane polymer (degree of polymerization with 4 to 20 repetition units), carboxy, silyl, alkyl silyl, alcoxy silyl, siloxy, alkyl siloxy, alkoxy siloxy, silyl alkyl, alkoxy silyl alkyl, alkyl silyl alkyl, halogen, epoxy (C2-C20), ester, aryl ether, fluoroalkyl, blocked isocyanate, acrylate, methacrylate, mercapto, nitrile, amine, and/or phosphine group, each substituted or unsubstituted.

The residues R may be the same or different.

Preferably, R is independently from each other: hydroxy, alkyl (C4-C10), aryl (C6-C12), especially phenyl and tolyl, alkoxyl (C4-C10), alkenyl (C2-C10), oxirane polymer, especially polyethylene glycol, polypropylene glycol, polybutylene glycol, and/or their copolymers (degree of polymerization 4 to 20, especially 10 to 15 repetition units), epoxy (C2-C10), and/or cycloalkyl (C5-C10), each substituted or unsubstituted.

Especially preferred, R is independently from each other: hydroxy, alkyl (C4-C10), phenyl, tolyl, alkoxyl (C4-C10), alkenyl (C2-C10) and/or oxirane polymer, especially polyethylene glycol, polypropylene glycol, and/or their copolymers (degree of polymerization 4 to 20, especially 10 to 15 repetition units), each substituted or unsubstituted.

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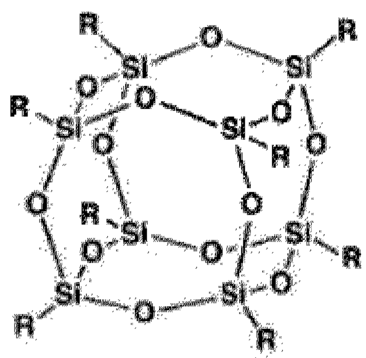
In another embodiment of the present disclosure, R may comprise additional functional groups, especially thiol groups, phosphate groups individually or in combination. The optionally present thiol or phosphate groups may additionally react with the metal surface to be protected.

In another preferred embodiment, the silasesquioxane may have a structure that is derived from the chemical formula $[RSiO_{3/2}]_n$ in which one or more, preferably one, unit of silicone RSi is replaced by other units. This silasesquioxane preferably has the formula $[RSiO_{3/2}]_n(R_2SiO)_3$ wherein the residue R may be selected independently from those described above and may be n = 2, 4, 6, 8, preferably n = 2, 4, 6 and especially 4.

The use of bridged molecular silasesquioxanes is conceivable as well.

In a particularly preferred embodiment, the silasesquioxane is a silasesquioxane according to formula (I):



(I)

wherein: R being independently from each other = oxirane polymer, preferably polyethylene glycol, polypropylene glycol, polybutylene glycol, and/or their copolymers (degree of polymerization 4 to 20, especially 10 to 15 repetition units) and especially $-CH_2CH_2(OCH_2CH_2)_mOCH_3$ and m = 10 to 15, especially 13.3,

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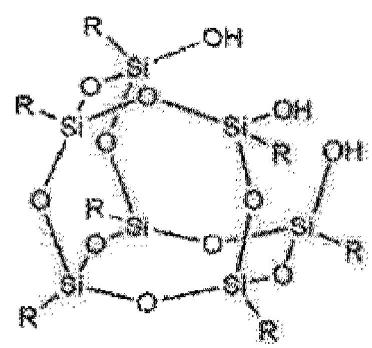
wherein the silasesquioxane may be present in the form of a mixture with other silasesquioxanes. Such a silasesquioxane is available in the form of a mixture with other silasesquioxanes for example under the tradename: PEG POSS® Cage Mixture by Hybrid Plastics.

In another preferred embodiment, the silasesquioxane is a silasesquioxane according to the formula (I) above wherein: R is independently from each other = alkyl (C4-C10), aryl (C6-C12), preferably isooctyl, isobutyl, and/or phenyl, especially isooctyl, wherein the silasesquioxane may be present in the form of a mixture with other silasesquioxanes. Such a silasesquioxane is available in the form of a mixture with other silasesquioxanes for example under the tradename: Isooctyl POSS® Cage Mixture and Octalsobutyl POSS® by Hybrid Plastics.

In a further, particularly preferred embodiment, the silasesquioxane is the silasesquioxane according to formula (II):



(II)

wherein: R being independently from each other = alkyl (C4-C10), preferably isooctyl. Such a silasesquioxane is available under the tradename: TriSilanolIsobutyl POSS® by Hybrid Plastics.

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It was found that the lubricant composition may also comprise mixtures of structurally different silasesquioxanes.

In a further preferred embodiment, the silasesquioxane is present on nanoparticulate carrier materials, preferably on oxidic nanoparticles, especially on amorphous silicone dioxide nanoparticles. These types of silasesquioxanes are available for example under the tradename POSS® Nanosilica Dispersion by Hybrid Plastics. What is advantageous about them is the very good stabilization of the nanoparticles in various media and the combination of the two different particle sizes.

The silasesquioxane may be mixed directly with the base oil of the lubricant or in the form of a premixture. In the case where it is introduced in the form of a premixture, said premixture preferably contains a carrier material, preferably selected from the group consisting of mineral oils, synthetic hydrocarbons, and from them preferably polyalphaolefins (PAO) and metallocene-catalyzed PAO (m-PAO), polyglycols, esters, perfluoropolyether (PFPE), silicone oils, virgin oils and derivatives of virgin oils, oils containing aromatic compounds such as phenyl ethers, alkylated naphthalenes and the mixture of the aforementioned carrier materials. An especially preferred carrier material contains polyglycols, ester, and synthetic hydrocarbons.

The base oil of the lubricant compositions is preferably selected from the group consisting of polyglycols, silicone oils, PFPE, mineral oils, esters, synthetic hydrocarbons, from among them preferably PAO, m-PAO, oils containing aromatic compounds such as phenyl ethers, alkylated diphenyl ethers, alkylated naphtalenes, phenyl ethers, virgin oils and derivatives of virgin oils, and the mixtures of the aforementioned base oils. Especially preferred base oils use

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polyglycols, esters, and/or synthetic carbon hydrogens, and from them especially preferred polyalphaolefins (PAO) and metallocene-catalyzed PAO (m-PAO). Especially preferred esters are selected from an ester of an aliphatic or aromatic di, tri, or tetra-carbonic acid (preferably C_6- to C_{60}-) with a or in mixture present C_7- to C_{22}- alcohols, from one ester of trimethylolpropane, pentaerythrite, or di-pentaerythrite with aliphatic C_7- to C_{22}- carbonic acids, from C_{18}-dimeric acid esters with C_7- to C_{22}-alcohols, from complex esters, as individual components or in any mixture.

The lubrication composition may furthermore contain other common additives such as thickeners (metal soaps, metal complex soaps, bentonite, ureas, silicates, sulfonates, polyimides, etc.), solid lubricants (PTFE, metal oxide, graphite, sulfonate, polyimide, etc.) and additives (phosphate, thiophosphate, aromatic amines, phenols, sulfates, etc.) Preferred thickeners are lithium soaps, lithium complex soaps, ureas, calcium complex soaps, calcium sulfonate thickeners, bentonite, aluminum complex soaps. Especially preferred thickeners are lithium soaps, lithium complex soaps, aluminum complex soaps, bentonite and ureas.

Said additives may be soluble additives especially as corrosion inhibitors, as a means to reduce friction, as a means to protect against metallic influences, and as UV stabilizers.

For transmission applications, it was found to be especially favorable if the lubricant composition has a viscosity of ISO VG 68-680, especially favorably ISO VG 220-460. The preferred base oils that are used are polyglycols on the one hand and mixtures of synthetic hydrocarbons on the other hand, and among them especially preferred mixtures of PAO with m-PAO, mixtures of esters or

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compositions that comprise mixtures of synthetic hydrocarbons and esters as base oils. Medicinal white oils are suitable as well.

For greased applications in wind turbines, it was found that it is especially favorable if the lubrication composition has an NLGI class according to DIN 51818 between 0 and 3, preferably 1 or 2. The base oil preferably has a viscosity ranging from 50 to 460 mm^2/sec. Preferred base oils are PAO, m-PAO, ester, and their mixtures. Preferred thickeners are lithium soaps, lithium complex soaps, and ureas.

For applications in the automotive sector, it was found to be especially favorable if the lubricant composition has an NLGI class according to DIN 51818 between 1 and 3. Preferred base oils are mineral oils, PAO, m-PAO, ester, and their mixtures. Preferred thickeners are lithium soaps, lithium complex soaps, calcium complex soaps, and ureas.

Here, the base oil preferably has a viscosity ranging from 30 to 300 mm^2/sec, and even more preferably from 50 to 200 mm^2/sec.

The lubricant composition contains the silasesquioxane preferably in quantities from 0.01 to 40 wt%, more preferably from 0.05 to 20 wt%, even more preferably in quantities from 0.07 to 15 wt%, and especially from 0.1 to 10 wt% relative to the total weight of the lubricant composition. In another preferred embodiment, the lubricant composition contains the silasesquioxane in quantities from 0.05 to 5 wt%.

In an embodiment, the lubricant composition may contain the base oil in quantities, for example, from 99.99 to 50 wt%, or from 99 to 60 wt%, or from 98 to 65 wt%, relative to the total weight of the lubricant composition. In an

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embodiment, the lubricant composition may contain base oil in a quantity of around 96.99 to 50 wt% relative to the total weight of the lubricant composition.

In a particularly preferred embodiment of the present disclosure, the lubricant composition of the present disclosure contains polyglycol as the base oil in combination with a silasesquioxane of the formula $[RSiO_{3/2}]_n$ with n = 6, 8, 10, 12, preferably n = 8, 10, 12 and especially 8, wherein R is independently from each other = oxirane polymer, especially polyethylene glycol, polypropylene glycol, and or their polymers (degree of polymerization 4 to 20, especially 10 to 15 repetition units).

Also conceivable is the combination of polyglycol as the base oil with a silasesquioxane of the formula $[RSiO_{3/2}]n(R_2SiO)_3$, with n = 2, 4, 6, 8, preferably n = 2, 4, 6 and especially 4, wherein R being independently from each other = alkyl (C4-C10), preferably isooctyl.

The combination of polyglycol as the base oil with PEGPOSS® Cage Mixture is especially preferred.

In a further, particularly preferred embodiment of the present disclosure, the lubricant composition contains ester, hydrocarbons, alkylated diphenyl ethers as the base oil in combination with a silasesquioxane according to the formula $[RSiO_{3/2}]_n$ with n = 6, 8, 10, 12, preferably n = 8, 10, 12 and especially 8, wherein R = alkyl (C1-C20) or aryl (C6-C18) and more preferably isooctyl, isobutyl, and/or phenol.

In another particularly preferred embodiment of the present disclosure, the lubricant composition contains ester, hydrocarbons, alkylated diphenyl ethers as the base oil in combination with a silasesquioxane according to formula (I) above, wherein: R = isooctyl or isobutyl and/or phenol. The combination of esters,

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hydrocarbons, alkylated diphenyl ethers as base oil with IsooctylPOSS® Case Mixture, PhenylPOSS® OctaIsobutylPoss® is especially preferred.

The composition according to the present disclosure generally contains silasesquioxane in quantities from 0.01 to 40 wt%, more preferably from 0.05 to 20 wt%, even more preferably in quantities from 0.07 to 15 wt%, and especially from 0.1 to 10 wt%; base oil in quantities from 99.99 to 50 wt%, or in quantities from 96.99 to 50 wt%,, more preferably in quantities from 99 to 50 wt%, even more preferably in quantities from 99 to 60 wt%, especially in quantities from 98 to 65 wt%; thickeners in quantities from 3 to 40 wt%, more preferably in quantities from 5 to 40 wt%, and especially in quantities from 7 to 25 wt%, as well as solid lubricants in quantities from 0 wt% to 30 wt%, more preferably in quantities from 0 to 20 wt%, and additives in quantities from 0 wt% to 15 wt%, more preferably in quantities from 0 to 10 wt%, and especially in quantities from 2 to 10 wt%, each time relative to the total weight of the lubricant composition.

In a preferred embodiment of the present disclosure, the lubricant composition contains:

- 5 to 80 wt%, preferably from 10 to 80 wt%, more preferably from 20 to 70 wt%, and especially from 25 to 60 wt% polyalkylene glycol, preferably selected from the group consisting of statistically distributed polyoxyethylene and/or polyoxypropylene units and/or other polyoxyalkylene modules, a block polymer from polyoxyethylene and/or polyoxypropylene units and/or other polyoxyalkylene modules, as base oil and/or

- 5 to 80 wt%, preferably from 10 to 80 wt%, more preferably from 20 to 70 wt%, and especially from 25 to 60 wt% carbonic acid ester as base oil and/or
- 2 to 80 wt%, preferably from 10 to 80 wt%, more preferably from 20 to 70 wt%, and especially from 25 to 60 wt% fatty alcohol ethoxylate as base oil,
- More than 10 wt%, preferably from 15 to 85 wt%, more preferably from 20 to 60 wt%, and especially from 25 to 50 wt% water,
- 0.01 to 40 wt%, more preferably from 0.05 to 20 wt%, even more preferably from 0.07 to 15 wt%, and/or from 0.05 to 5 wt% and/or from 01. to 10 wt% silasesquioxane,

with the quantities provided being relative to the total weight of the lubricant composition.

Due to the water component, this lubricant composition can be considered a water-based lubricant composition.

In a preferred embodiment of the present disclosure, the lubricant composition is present as a water-based transmission oil formulation with which, during the performance of an FZG test according to DIN ISO 14635-3, the load stage 12 is passed with a total wear of the gear and pinion being < 150 mg, and preferably, after a subsequent longer 50-hour test at load stage 10, no significant further wear is generated.

Preferred base oils for the water-based lubricant composition are water-soluble polyalkylene glycols, water-soluble carbonic acid esters, and/or water-soluble fatty alcohol ethoxylates. According to the present disclosure, the term “water-soluble” means that a transparent liquid is present after the base oils have been

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mixed with water (stirred for one hour) in a concentration ratio of at least 5 wt% base oil in water at room temperature (25°C).

Especially preferred carbonic acid ester base oils for the water-based lubricant composition are selected from the group consisting of ethoxylated mono- or di-carbonic acids with a chain length from C_4- to C_{40}- and ethoxylation degrees of 2-15.

Preferred fatty alcohol ethoxylates consist of fatty alcohols with chain lengths from C_6- to C_{22}- and an ethoxylation degree of greater than 3.

Preferred additives for the water-based lubricant composition are selected from the group consisting of:

- 0.5 to 20 wt%, preferably from 0.5 to 10 wt%, foaming or non-foaming emulsifiers of the class of the anionic, non-anionic, or cationic surfactants, preferably selected from the group consisting of aliphatic or aromatic ethoxylates, carboxylates, sulfonates, sulfates, or ammonium salts,
- 0.5 to 50 wt%, preferably from 1 to 10 wt%, antifreeze agents, selected from the group consisting of alkylene glycol, glycerin, or ionic fluids,
- 0.5 to 20 wt%, preferably from 5 to 20 wt%, corrosion additives, selected from the group consisting of alkanolamines, phosphoric acid, and carbonic acid derivatives,
- 0.001 to 2 wt%, preferably from 0.01 to 1 wt%, additives to prevent the development of foam, selected from the group consisting of polydimethylsiloxane and acrylate polymers,

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- 0.05 to 10 wt%, preferably from 1 to 5 wt%, water-soluble corrosion and wear protection agents, selected from the group consisting of sulfur or phosphorous-containing compounds,
- 0.001 to 0.5 wt%, preferably from 0.05 to 0.4 wt%, biocides, selected from the group consisting of substituted isothiazolinones and bronopol,
- and mixtures thereof.

In a further preferred embodiment of the present disclosure, the water-based lubricant composition contains 0.5 to 40 wt% lubricant thickener, selected from the group consisting of metallic soaps from mono- and/or di-carbonic acids, ureas, sheet silicates, solid lubricants, and Aerosil.

In a preferred embodiment of the present disclosure, the lubricant composition is present as a transmission oil formulation with which during the performance of an FZG gray staining test C/8.3/60 according to the FVA information sheet 54/7 with injection lubrication, the profile deviation does not exceed 7.5 µm during the stepped test and/or 20 µm during the continuous test.

In a preferred embodiment of the present disclosure, the lubricant composition is characterized by the fact that, during the performance of a false Brinell test using an SNR FEB 2 test device at room temperature, 8000 N load, a 3° pivoting angle, and a 24 Hz oscillation frequency, a run time of at least 50 hours is achieved and the wear of the drive element is preferably below 100 mg, especially below 20 mg.

In another preferred embodiment of the present disclosure, the lubricant composition is characterized by the fact that a mass loss of the drive element

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caused by vibrations is reduced by at least 50%, preferably by at least 90%, and/or the time to failure is at least doubled.

The present disclosure also relates to the use of the lubricant composition according to the present disclosure for the treatment of surfaces of drive elements, preferably rolling bearings, transmissions, friction bearings, and/or chains, especially rolling bearings and transmissions. The lubricant composition according to the present disclosure is also suitable for the lubrication of seals on rotating shafts.

Its use is especially advantageous in rolling bearings that are used as wheel bearings and/or transmissions that are exposed to vibration. Its use is furthermore advantageous in main bearings, blade bearings, adjusting bearings, and generator bearings of wind turbines. Its use is especially advantageous in rolling bearings that are used in electric motors of electrically driven vehicles. Its use is especially advantageous in rolling bearings in clutches, especially in hybrid vehicles. Its use is furthermore especially advantageous in bearings in ancillary components both in industrial installations as well as in motor vehicles. Bearings in ancillary components are characterized by the fact that the ancillary components are generally not in continuous operation but are only switched on temporarily, which means that vibrations act on the stationary bearings. Ancillary components in motor vehicles are furthermore often driven by pulleys. Furthermore, especially advantageous is the use of the lubricant in joints in automotive applications such as universal joints, Azipod joints, tripod joints, chassis joints, and/or ball joints which are also known for material fatigue/blowouts as the failure mode.

The aforementioned drive elements are particularly susceptible to the damage mechanisms described above so that the use of silasesquioxanes with their advantageous influence on the same is particularly efficient.

Especially preferred is furthermore the treatment of surfaces of drive elements in machines and conveyor systems that are used for the production of food where direct contact of the lubricant composition with the food is possible and a corresponding permit according to food law is required (USDA or NSF, kosher, halal).

Another aspect of the present disclosure relates to a use of drive elements, preferably rolling gears, transmission, friction gears, and/or chains whose surfaces were treated with the lubricant composition according to the present disclosure, for example in installations and machines for the production and conveyance of food, in wind turbines, in motor vehicles, in pulley gears, in rail vehicles, in ships, in electric motors, generators, ancillary components, and joints.

Descriptions in the present disclosure of features and/or embodiments as being “preferred”, “more preferred”, “even more preferred”, “especially preferred” “preferably”, “advantageous”, “especially advantageous”, etc., are not limiting in any way. Rather, these descriptions merely describe example features and/or embodiments according to the present disclosure. For example, an embodiment or feature is not necessarily preferred or advantageous in any or all situations or embodiments.

The structure, features, accessories, and alternatives of specific embodiments described herein are intended to apply generally to all of the teachings of the present disclosure, including to all of the embodiments described and illustrated herein, insofar as they are compatible. In other words, the structure, features,

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accessories, and alternatives of a specific embodiment are not intended to be limited to only that specific embodiment unless so indicated.

The embodiments according to the present disclosure are intended to be examples only. Alterations, modifications and variations may be effected to the particular embodiments by those of skill in the art without departing from the scope, which is defined solely by the claims appended hereto.

Test Methods Used:

The SNR-FEB 2 (false brinelling test device made by SNR, a company that makes rolling gears) determines the wear behavior of lubricant compositions in rolling gears during small, oscillating rolling and sliding movements under constant load. The switch-off criterion of the SNR is the wear progression. If the value increases in a bearing above 30 mm or if the predetermined run time is reached, the run is automatically ended. The bearing type FAG 51206 is used as the test bearing. The resulting wear is not determined on the basis of the wear progression, however, but by weighing the cleaned bearing rings before and after the test. The grooves in the bearing rings are completely filled with the lubricant composition to be tested and excess fat wiped off. Depending on the density, approx. 1 g of the lubricant composition is used for each bearing ring.

Flender Foam Test GG-V 425 Rev. 1

The test device consists of a closed gearbox housing with a window. Two gears of the same size (outer diameter 54 mm) are centrally placed over vertically

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positioned shafts that dip into the test oil so that part of the gears is not covered by oil. At a revolution speed of 1450 rpm, the pair of gears is driven for 5 minutes. This causes air to be mixed into the oil. The change/increase in volume can be documented due to a scale that is applied to the window. The threshold values of the standard are: 1 min after the discontinuation of the operation of the pair of gears $\leq$ 15% and $\leq$ 10% total after 5 minutes. The foam volumes may not be exceeded.

Viscosity

Viscosity measurement (DIN 51562) by using a Stabinger viscosimeter SVM 3000 (Anton Paar).

Foam Test ASTM D 892

In this method, air is foamed with a constant volume stream by means of an immersed sinter ball at room temperature, then at 94°C, and then at room temperature again for one minute. It measures a) how much foam is generated in ml and b) how long it takes for the foam to disintegrate again after air is no longer introduced. The results are provided (a,b). The threshold value: (max. 75 ml/10 min) may not be exceeded in any of the three temperature sequences. b is provided in the form x:y min. That means that the foam disintegrated again after x minutes and y seconds.

Gray Staining Test

C/8.3/60 according to the FVA information sheet 54/7 with injection lubrication. The threshold values of the standard are: the profile deviation does not exceed

7.5 µm during the stepped test and/or 20 µm during the continuous test. The test was, in parts, performed in deviation at 90°C and immersion lubrication as well.

FZG test A/2.8/50 for the determination of the relative abrasion capacity and wear behavior of semi-fluid transmission greases

Performed according to the standard DIN ISO 14635-3 with submersion lubrication. The threshold values of the standard are:

Load stage reached = total of all damage (width of all marks and abrasion) on the active tooth flanks of the 16 pinion teeth is more than the width of a tooth or 20 mm.

Additional analysis of wear on the gear and pinion.

Additionally, after the end of the test, a long test for a period of 50 hours was performed at load stage 10 under determination of the wear on the gear and pinion.

Filtration Test

A heatable oil reservoir (60°C) is filled with approx. 10 L oil. With a controllable pump (made by Vogel Fluidtec GmbH / flow sensor controlled), the oil is pumped in a circle (6 L/min) through a filter with a precisely defined pore size (Mahle Pl 2105 PS 3 µm/ Mahle Pl 3105 PS 10 µm). Sensors measure the pressure in front of and behind the filter. The system shuts off if the pressure difference exceeds 2.2 bar. The length of the test may be up to 840 h.

Example 1: **Test for the improvement of the false Brinell protection**

A lithium soap fat of the NLGI class 2 with polyglycol base oil with a viscosity of approx. 46 mm^2/sec at 40°C and additive packet (corrosion, oxidation stability, load-bearing capacity, wear) was mixed with 5.85% PEGPOSS® Cage Mixture and homogenized with a speed mixer (by Hauschild, type DAC 700.1 FVZ) (exemplary fat 1). PEGPOSS® Cage Mixture has a viscosity of approx. 80 mm^2/sec at 40°C. To compensate for the dilution effect, a comparison fat 1 was produced in which the fat 5.85% of a polyglycol on the basis EO:PO approx. 1:1 with comparable viscosity was diluted and homogenized in the same way. Both fats were exposed in the SNR FEB 2 test device to a load of 8000 N, a pivoting angle of 3°, and a 24 Hz oscillation frequency at 20°C. The exemplary fat 1 reached the intended test duration of 50 h with just a low mass loss of the bearings. The comparison fat 1, however, reached the maximum permitted wear after approx. 19 h and the test had to be terminated.

	Exemplary fat 1, PG fat, lithium soap, PG base oil + diluted with 5.85% PEGPOSS® Cage Mixture	Comparison fat 1, PG fat, lithium soap, PG base oil + diluted with 5.85% PG base oil
Wear bearing 1 (mg)	21	95
Wear bearing 2 (mg)	0	216
Test duration	50 h	19 h, 28 min (termination)

Example 2: Effect of silasesquioxane for the suppression of gray staining in an ester oil for transmissions

	Reference oil 2: Transmission oil on ester basis, ISO VG 100 additive package (corrosion, foam, oxidation	Reference oil 2 + 1.3% isooctyl POSS® Cage Mixture

	stability, load-bearing capacity, wear)	
Kinematic viscosity 40°C [mm^2/sec]	98	99.3
ASTM Foam Test		
RT	0 ml / 0 min	0 ml / 0:0 min
94°C	0 ml / 0 min	0 ml / 0:0 min
RT	0 ml / 0 min	10 ml / 0:03 min
FZG gray staining test, C/8.3/60		
Stepped test, weight change (mg) after load stage 10	24	16
Stepped test, gray staining surface (%) after load stage 10/400 h	30	20
Stepped test, profile form deviation (µm) after load stage 10/400 h	8.8 (fail)	7.3 (pass)
Continuous test, weight change (mg) after load stage 10/400 h	fail*	35
Continuous test, gray staining surface (%) after load stage 10	fail*	23
Continuous test, profile form deviation (µm) after load stage 10	fail*	10.8 (pass)
*Failure after 240 h load stage 10 for exceeding the damage threshold, profile form deviation > 20 µm		

The results show that no changes occurred in the kinematic viscosity and the foaming behavior of the transmission oil on ester basis when adding IsooctylPOSS®. The development of gray staining is significantly suppressed, however. The addition of IsooctylPOSS® Cage Mixture makes it possible to perform the test for the duration of the test and for the profile form deviation to remain significantly below the thresholds.

Example 3: Effect of silasesquioxane for the reduction of foam and gray staining in polyglycol-based transmission oil

	Reference oil 3: Transmission oil polyglycol, additive package (corrosion, oxidation stability, load-bearing capacity, wear, foam) ISO VG 100	Reference oil 3 + 5.8% PEGPOSS® Cage Mixture
Viscosity 40°C [mm^2/sec]	101	102
ASTM Foam Test		
RT	10 ml / 0:15 min	0 ml / 0:0 min
94°C	20 ml / 0:15 min	10 ml / 0:0 min
RT	20 ml / 0:25 min	10 ml / 0.03 min
Flender Foam Test		
Foam after 1 min rest (%)	20	0
Foam after 5 min rest (%)	19	4
FZG gray staining test, C/8.3/90°C immersion lubrication		
Stepped test, weight change (mg) after load stage 10	17	1
Stepped test, gray staining surface (%) after load stage 10	10	0
Stepped test, profile form deviation (µm) after load stage 10	5	4.6
Continuous test, weight change (mg) after load stage 10/400 h	11	8
Continuous test, gray staining surface (%) after load stage 10/400 h	20	0
Continuous test, profile form deviation (µm) after load stage 10/400 h	9.6	7

There were no significant changes in the viscosity and ASTM foam test when mixing in PEGPOSS® Cage Mixture. A significant improvement is seen, however, in the Flender foam test, and the development of gray staining is suppressed almost completely.

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Example 4: Filterability of oils containing silasesquioxane in comparison with oils containing SiO_2 nanoparticles

	Reference oil 4: Transmission oil PAO/ester ISO VG 46 Additive package (corrosion, oxidation stability, load-bearing capacity, wear, foam)	Reference oil 4 + 1.3% IsooctylPOSS®	Reference oil 4 + 0.5% SiO_2 nanoparticles 10 nm, surface coated with phenyl and trimethyl silyl groups
Viscosity 40°C [mm^2/sec]	46.2	47	47.6
Flender Foam Test			
Foam after 1 min rest (%)	2	2	2
Foam after 5 min rest (%)	0	3	2
Filtration test 3 µm filter			
Pressure buildup/test duration	<2.2 bar/840 h	<2.2 bar/840 h	>2.2 bar/0 h
Filterability	pass	pass	fail
Flender Foam test, after filtration test 3 µm			
Foam after 1 min rest (%)	4	5	Undetermined
Foam after 5 min rest (%)	5	5	Undetermined
Filtration test 10 µm filter			
Pressure buildup/test duration	Undetermined	<2.2 bar/760 h	>2.2 bar/0 h
Filterability	Undetermined	pass	fail

The reference oil 4 can easily be filtered at 3 µm. Adding IsooctylPOSS® Cage Mixture had no effect on the viscosity and the good filtration and foam behavior.

If SiO_2 nanoparticles, which can also be used to reduce gray staining, are used, however, high pressures are required for an effective filtration. The part of inorganic SiO_x is approximately the same in the two oils with the additives containing silicone.

Even if using a coarser filter with 10 µm, it is not possible to filter the oil containing SiO_2 at lower pressures. A pressure of over 2.2 bar builds up immediately, and the test is terminated. The oil containing the IsooctylPOSS® Cage Mixture, however, can be filtered without any problem.

Example 5: Effect of silasesquioxane for the reduction of wear in water-based transmission oil

	Reference oil 5: water-based transmission oil on polyglycol basis (approx. 39 wt%) Additive package (approx. 21 wt% corrosion, load-bearing capacity/wear, foam, biocide), water (approx. 40 wt%)	Reference oil 5 + 1 wt% PEGPOSS® Cage Mixture
Viscosity 40°C	ISO VG 460	ISO VG 460
FZG DIN ISO 14635-3-A/2.8/50 Submersion Lubrication		
Load stage achieved	12	12
Overall wear after load stage 11	188 mg	96 mg
Overall wear after load stage 12	231 mg	145 mg
Additional wear after load stage 10/50 h	63 mg	7 mg

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By adding PEGPOSS® Cage Mixture, no significant change in terms of viscosity was found, but the wear behavior in transmission applications, especially under moderate load (load stage 10), improved significantly.

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Claims:

1. A lubricant composition for application onto a surface of drive elements, the lubricant composition comprising:

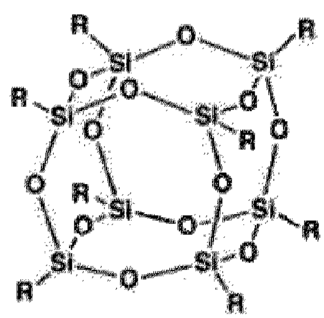
a base oil; and

a silasesquioxane;

wherein the silasesquioxane has chemical formula $[RSiO_{3/2}]_n$, wherein n = 6, 8, 10, or 12, and R comprises an oxirane polymer with a degree of polymerization of 4 to 20 repetition units.

2. The lubricant composition for application onto a surface of drive elements according to claim 1, wherein, the oxirane polymer comprises one or more of polyethylene glycol, polypropylene glycol, polybutylene glycol, and copolymers thereof, and wherein the oxirane polymer has a degree of polymerization of 10 to 15 repetition units.

3. The lubricant composition for application onto a surface of drive elements according to claim 1 or 2, wherein the silasesquioxane comprises a silasesquioxane according to formula (I):



(I)

wherein the oxirane polymer comprises one or more of polyethylene glycol, polypropylene glycol, polybutylene glycol, and copolymers thereof,

wherein the oxirane polymer has a degree of polymerization of 10 to 15 repetition units.

4. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-3, wherein the silasesquioxane is present on nanoparticulate carrier materials.

5. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-4, wherein the base oil comprises one or more of polyglycols, silicone oils, PFPE, mineral oils, esters, synthetic hydrocarbons, oils containing aromatic components, virgin oils, and derivatives of virgin oils.

6. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-5, wherein the lubricant composition comprises:

- at least one of:

  5 to 80 wt% polyalkylene glycol as base oil;

  5 to 80 wt% carbonic acid ester as base oil; and

  2 to 80 wt% fatty alcohol ethoxylate as base oil,

- More than 10 wt% water; and

- 0.01 to 40 wt% silasesquioxane,

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wherein each amount is based on a total weight of the lubricant composition.

7. The lubricant composition for application onto a surface of drive elements according to claim 6, wherein at least one of the polyalkylene glycol, the carbonic acid ester, and the fatty alcohol ethoxylate is water soluble.

8. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-5, wherein the silasesquioxane is present in an amount of from 0.01 to 40 wt% relative to a total weight of the lubricant composition.

9. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-8, wherein the base oil comprises polyglycol.

10. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-9, wherein:

silasesquioxane is present in an amount of from 0.01 to 40 wt%, base oil is present in an amount of from 96.99 to 50 wt%, and thickeners are present in an amount of from 3 to 40 wt%, and each amount is based on a total weight of the lubricant composition.

11. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-10, wherein the lubricant composition comprises a transmission oil formulation, and wherein, during a performance of an FZG gray staining test C/8.3/60 according to FVA information sheet 54/7 with injection lubrication, at least one of the following occurs:

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a profile deviation does not exceed 7.5 µm during a stepped test; and

the profile deviation does not exceed 20 µm during a continuous test.

12. The lubricant composition for application onto a surface of drive elements according to any one of claims 1-11, wherein during a performance of a false Brinell test using an SNR FEB 2 test device at room temperature, 8000 N load, a 3° pivoting angle, a 24 Hz oscillation frequency, and a run time of at least 50 hours, the wear of the drive element is below 100 mg.

13. The lubricant composition for application onto a surface of drive elements according to any one of claims 1 to 12, wherein the oxirane polymer comprises $-CH_2CH_2(OCH_2CH_2)_mOCH_3$, wherein m is 10 to 15.

14. A use of the lubricant composition as defined in any one of claims 1-13 for a treatment of surfaces of drive elements.

15. A use of drive elements comprising one or more of rolling bearings, transmissions, friction bearings, and chains, wherein surfaces of the drive elements have been treated with the lubricant composition as defined in any one of claims 1-13, the use comprising:

using the drive elements in installations and machines configured to produce and convey foodstuffs, in wind turbines, in motor vehicles, in pulley gears, in rail vehicles, in ships, in electric motors, generators, ancillary components, or joints.

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