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(54) **LUBRICATING GREASE COMPOSITION**

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(57) **ABSTRACT**

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A lubricating grease composition contains a major amount of lubricating base oil, at least one thickener and at least one agent for reducing the coefficient of friction. The agent for reducing the coefficient of friction comprises a metal oxide that has a specific surface area of at least 100 m²/g and that is selected from the group consisting of titanium oxide, silicon dioxide, aluminum oxide and tin dioxide.

LUBRICATING GREASE COMPOSITION

TECHNICAL FIELD

[0001] The present invention relates to a lubricating grease composition containing a major amount of lubricating base oil, at least one thickener and at least one agent for reducing the coefficient of friction. The lubricating grease composition according to the invention is particularly well-suited for use in tribological systems consisting of steel and plastic such as bearing guides and joints in the chassis of motor vehicles.

BACKGROUND OF THE INVENTION

[0002] Lubricating grease is used in the most various machine elements such as roller bearings, sliding bearings and wheel bearings, joints or gears. They normally consist of a synthetic oil or mineral oil as the lubricating base oil as well as of a soap as the thickener. Moreover, in order to adapt the lubricating grease to the specific application purpose, they can also contain various additives for improving the structural and boundary lubrication properties, the rheological and chemical properties as well as the appearance or odor. The composition and properties of conventional lubricants and greases are disclosed, for example, in Ullmanns Enzyklopädie der Technischen Chemie [Ullmann's Encyclopedia of Industrial Chemistry], 4th edition, Volume 20 (1981), pp. 460-671. Reference is hereby made to this disclosure.

[0003] Lubricating grease for use in joints and other bearing guides is normally made of a mineral oil and of a complex soap containing lithium and barium. However, smaller construction spaces for the bearing guides and as well as customer-specific requirements for greater stiffness of the joints lead to smaller friction surfaces and to the occurrence of higher surface pressures. Consequently, the lubricating grease has to have a generally lower coefficient of friction and must be able to withstand high loads. Conventional lubricating grease on the basis of mineral oil no longer meets these requirements. Moreover, the use of soaps containing barium, which has been widespread until now, is raising toxicological concerns, thus making it more difficult to return and recycle the lubricating grease.

[0004] The lubricating grease proposed as an alternative on the basis of polyureas and mineral oil or polyglycols and lithium soaps of the type known, for example, from European Patent No. 0 523 064 B1, however, have a poor elastomer compatibility and only provide insufficient corrosion protection.

[0005] Therefore, there is still a need for lubricating grease that can withstand the high loads that occur in joints and other bearing guides and whose use can prolong the service life of the joints and bearing guides.

BRIEF SUMMARY OF THE INVENTION

[0006] According to the invention, a lubricating grease composition contains a major amount of lubricating base oil, at least one thickener and at least one agent for reducing the coefficient of friction. The agent for reducing the coefficient of friction comprises a metal oxide that has a specific surface area of at least 100 m²/g and that is selected from the group consisting of titanium oxide, silicon dioxide, aluminum oxide and tin dioxide.

[0007] In studying the insufficient corrosion protection of lubricating grease on the basis of polyalkylene glycols and lithium soaps in tribological systems consisting of steel and plastic, a high amount of the polyoxymethylene (POM) used as the bearing shell material could be found in the lubricating grease. It was also found that an additional input of energy into the lubricating grease, for example, during prolonged times of exposure to hot environment or after renewed high stress on the joint, led to autocatalytic oxidation of the lubricant caused by the plastic abrasion and thus ultimately to the failure of the lubrication and to the corrosion of the joint.

[0008] Concurrent measurements of the coefficient of friction have also ascertained that the lubricants known so far exhibit a more or less pronounced scattering of the coefficients of friction over the duration of the experiment. The peaks in the coefficient of friction that occurred here were recognized as the cause of the increased plastic abrasion. Moreover, an appreciable amount of metal particles could be detected in the examined lubricating grease on the basis of polyalkylene glycol and lithium soap. This indicated that the examined lubricating grease could not be considered to be low-friction vis-à-vis steel either.

[0009] The experiments performed led to the realization that, in order to improve the corrosion protection and the service life of the joints, it was desirable to reduce and stabilize the coefficient of friction of the lubricating grease composition, thus reducing the plastic abrasion that preceded the corrosion.

[0010] Surprisingly, it was found that the addition of metal oxides from the group consisting of titanium oxide, silicon oxide and tin oxide having a specific surface area of at least 100 m²/g reduces the coefficients of friction of the examined lubricating grease compositions and additionally leads to coefficients of friction that remain constant over the duration of the experiment.

[0011] According to the invention, the metal oxide can be added in an amount of 0.1% to 5% by weight. If larger amounts are added, then an undesired thickening effect is expected to occur. Smaller amounts do not have any considerable influence on the coefficients of friction. It is particularly preferred that the metal oxide is present in an amount of 0.5% to 2.5% by weight.

[0012] According to the invention, the specific surface area of the metal oxide is at least 100 m²/g. With smaller specific surface areas, the distribution of the metal oxide in the lubricating grease is insufficient and an increase in the coefficients of friction can occur. Preferably, the specific surface area of the metal oxide is at least 150 m²/g, particularly preferably at least 200 m²/g.

[0013] In a particularly preferred embodiment of the invention, the metal oxide is a highly dispersed (fine-particle-size) silica (Aerosil) that can advantageously be hydrophobized and that has a specific surface area of between 200 m²/g and 300 m²/g. Such silicas are commercially available.

[0014] The base oil of the lubricating grease composition according to the invention can be selected from the group consisting of mineral oil, synthetic oils and their mixtures. Examples of suitable synthetic oils are in particular polyalpha-olefins, polyesters and polyalkylene glycols. Particularly preferably, the base oil is a polyalkylene glycol, in particular polypropylene glycol, optionally in a mixture with small fractions of ester oils, in particular esters of neopentyl

polyols, and synthetic hydrocarbons such as alkyl benzenes. The base oil fraction is preferably in the range between 40% and 90% by weight, relative to the total weight.

[0015] The known soaps of alli and earth alkali metals as well as aluminum can be used as the thickener. Simple or complex lithium soaps of the type known, for example, from EP 523 064 B1 (incorporated herein by reference), are particularly preferred. Special preference is given to complex lithium soaps of C₁₂-C₂₄ hydroxycarboxylic acids, in particular 12-hydroxy stearic acid, in a mixture with a C₂-C₁₂ dicarboxylic acid such as, for example, azelaic acid and/or sebacic acid. The fraction of the thickener is preferably in the range between 5% and 30% by weight, relative to the lubricating grease composition, particularly preferably at least 7% by weight.

[0016] The lubricating grease composition can also contain further known additives such as, for example, antioxidants, viscosity-index improvers, pour point depressing additives, coefficient of friction reducers and anticorrosion agents, preferably in an amount of up to 35% by weight.

[0017] Finally, the invention also relates to the use of a metal oxide from the group consisting of silicon dioxide, titanium oxide, tin dioxide and aluminum oxide having a specific surface area of at least 100 m²/g as an agent in a method of reducing and stabilizing the coefficient of friction of lubricating grease, in particular in tribological systems consisting of steel and plastic.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[0018] The lubricating grease composition according to the invention stands out for its considerably reduced coefficients of friction which also exhibit a small range of fluctuation. As a result, the abrasion of plastic and metal particles from the joint shells and the joint head is reduced, the energy applied into the lubricating grease is reduced and the risk of autocatalytic oxidation of the lubricating grease is reduced, consequently improving the corrosion protection and the service life of the joints. Subsequent lubrication of the joints and bearing guides lubricated with the composition according to the invention are thus superfluous.

[0019] In particular, lubricating grease with the following mechanical and chemical characteristic values has proven to be advantageous for use in chassis joints:

Base oil viscosity V ₄₀	≥300 mm ² /s
Pour point	<243 K
Viscosity index of the base oil	<200
Dropping point	>220° C. [428° F.]
Range of application	-35° C. to 120° C. [-31° F. to 248° F.]

[0020] The lubricating grease compositions according to the invention are preferably in the NLGI consistency class 1-2 according to standard DIN 51818 and have a worked penetration according to DIN ISO 2137 of 290 to 310×0.1 mm.

[0021] The invention is now described with reference to the following examples, although not being limited to these.

EXAMPLE OF AN EMBODIMENT

[0022] A lubricating grease composition having the components listed below was produced. The figures in % by weight refer to the total composition.

Lubricating base oil	
Polypropylene glycol ⁽¹⁾	46.5% by weight
Trimethylol propane ester ⁽²⁾	13.5% by weight
Alkyl benzene ⁽³⁾	5.0% by weight
Thickener	
Lithium hydroxide	1.5% by weight
12-hydroxy stearic acid	5.0% by weight
Azelaic acid	1.5% by weight
Agent for reducing the coefficient of friction	
Highly dispersed silica (hydrophobized) ⁽⁴⁾	2.0% by weight
Other additives	
Oleic acid amide	5.0% by weight
Ethylene diamine stearate	5.0% by weight
Disodium sebacate	2.0% by weight
Poly(dihydrotrimethyl quinoline)	3.0% by weight
Antioxidants	10.0% by weight

⁽¹⁾available as Polyglykol B01/240 from Hoechst

⁽²⁾available as Priolube 3999 from Unichema

⁽³⁾available as Medialub 1350 from Kettlitz Chemie

⁽⁴⁾available as Aerosil 974 from Degussa

[0023] The liquid lubricating base oils and the components of the thickener, a complex lithium soap, were combined, melted and heated for about 1 hour to at least 130° C. [266° F.]. Then the mixture was heated further and the agent for reducing the coefficient of friction as well as the other additives were added at temperatures between 165° C. [329° F.] and 80° C. [176° F.]. Subsequently, the grease was ground in a conventional colloid mill.

[0024] The grease produced in this manner exhibited an anticorrosive degree of ≤2, measured in accordance with DIN 51802, and an oxidation resistance of <0.1 bar, measured in accordance with DIN 51808 at 100° C. [212° F.] for 200 hours. The lubricating grease according to the invention is suitable for use within the temperature range from -35° C. to 120° C. [-31° F. to 248° F.].

1. A lubricating grease composition containing a major amount of lubricating base oil, at least one thickener and at least one agent for reducing the coefficient of friction, said agent for reducing said coefficient of friction comprising a metal oxide that has a specific surface area of at least 100 m²/g and that is selected from the group consisting of titanium oxide, silicon dioxide, aluminum oxide and tin dioxide.

2. The composition according to claim 1, wherein said metal oxide is present in an amount of 0.1% to 5% by weight.

3. The composition according to claim 1, wherein said metal oxide is present in an amount of 0.5% to 2.5% by weight.

4. The composition according to claim 1, wherein said specific surface area is at least 150 m²/g.

5. The composition according to claim 4, wherein said specific surface area is at least 200 m²/g.

6. The composition according to claim 1, wherein said metal oxide is a highly dispersed silica.

7. The composition according to claim 6, wherein said highly dispersed silica is hydrophobized.

8. The composition according to claim 6, wherein said highly dispersed silica has a specific surface area of between 200 m²/g and 300 m²/g.

9. The composition according to claim 1, wherein said base oil is selected from the group consisting of mineral oil, synthetic oils and mixtures thereof.

10. The composition according to claim 9, wherein said base oil is a synthetic oil on the basis of a polyalkylene glycol.

11. The composition according to claim 10, wherein said base oil comprises polypropylene glycol, trimethylol propane ester and alkyl benzene.

12. The composition according to claim 1, wherein said thickener is a lithium soap in an amount of 2% to 30% by weight.

13. The composition according to claim 12, wherein said thickener comprises said lithium soap of 12-hydroxy stearic acid and azelaic acid.

14. The composition according to claim 1, comprising additional additives from the group of plasticizers, modifiers of the coefficient of friction, anticorrosion agents and anti-oxidants.

15. A method of reducing and stabilizing the coefficient of friction of lubricating grease wherein the lubricating grease is provided with a metal oxide selected from the group consisting of silicon dioxide, titanium oxide, tin dioxide and aluminum oxide having a specific surface area of at least 100 m²/g.

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