GREASE COMPOSITION FOR USE IN CONSTANT VELOCITY JOINTS
COMPRISING AT LEAST ONE TRI-NUCLEAR MOLYBDENUM COMPOUND

Inventors: Jisheng E, Hennef (DE); Frank Reher, Sieburg (DE); Stefanie Rosenkranz, Wachtberg (DE)

Correspondence Address:
GKN Driveline/TTG
c/o Kristin L. Murphy, 39533 Woodward Avenue, suite 140
Bloomfield Hills, MI 48304 (US)

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ABSTRACT
To solve the problem to provide for a new grease composition giving low wear and low friction primarily to constant velocity joints, a grease composition is suggested comprising a) a base oil composition; and b) 0.25% by weight to 5% by weight of at least one tri-nuclear molybdenum compound of the formula Mo₅Sk₉Q, wherein L are independently selected ligands having organo groups with a sufficient number of carbon atoms to render the compound soluble or dispersible in the oil, n is from 1 to 4, k varies from 4 through 7, Q is selected from the group of neutral electron donating compounds such as amines, alcohols, phosphines, and ethers, and z ranges from 0 to 5 and includes non-stoichiometric values.

Friction coefficient

Wear (µm/m)

Grease Samples

Grease Samples
FIG. 5A

FIG. 5B
GREASE COMPOSITION FOR USE IN CONSTANT VELOCITY JOINTS COMPRISING AT LEAST ONE TRI-NUCLEAR MOLYBDENUM COMPOUND

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application is a continuation of International Application No. PCT/EP2006/009716 filed Oct. 7, 2006 which is hereby incorporated by reference in its entirety.

FIELD

[0002] The present invention relates to a lubricating grease which is intended primarily for use in constant velocity universal joints, especially ball joints or tripod joints, which are used in the drivelines of motor vehicles.

BACKGROUND

[0003] The motions of components within constant velocity joints (CVJ) are complex with a combination of rolling, sliding and spinning. When the joints are under torque, the components are loaded together which can not only cause wear on the contact surfaces of the components, but also rolling contact fatigue and significant frictional forces between the surfaces. The wear can result in failure of the joints and the frictional forces can give rise to noise, vibration and harshness (NVH) in the driveline. NVH is normally “measured” by determining the axial forces generated in plunging type CVJ. Ideally the greases used in constant velocity joints need not only to reduce wear, but also have to have a low coefficient of friction to reduce the frictional forces and to reduce or prevent NVH.

[0004] Constant velocity joints also have sealing boots of elastomeric material which are usually of bellows shape, one end being connected to the outer part of the CVJ and the other end to the interconnecting or output shaft of the CVJ. The boot retains the grease in the joint and keeps out dirt and water.

[0005] Not only must the grease reduce wear and friction and prevent the premature initiation of rolling contact fatigue in a CVJ, it must also be compatible with the elastomeric material of which the boot is made. Otherwise there is a degradation of the boot material which causes premature failure of the boot, allowing the escape of the grease and ultimately failure of the CVJ. The two main types of material used for CVJ boots are polychloroprene rubber (CR) and thermoplastic elastomer (TPE), especially ether-ester block co-polymer thermoplastic elastomer (TPC-ET).

[0006] Typical CVJ greases have base oils which are blends of naphthenic (saturated rings) and paraffinic (straight and branched saturated chains) mineral oils. Synthetic oils may also be added. It is known that base oils have a large influence on the deterioration (swelling or shrinking) of both base oils of CR and TPC-ET. Both mineral and synthetic base oils extract the plasticisers and other oil soluble protective agents from the boot materials. Paraffinic mineral oils and poly-α-olefins (PAO) synthetic base oils diffuse very little into especially bases made of rubber material causing shrinkage, but on the other hand naphthenic mineral oils and synthetic esters diffuse into boot materials and act as plasticisers and can cause swelling. The exchange of plasticiser or plasticiser compositions for the naphthenic mineral oil can significantly reduce the boot performance, especially at low temperatures, and may cause the boot to fail by cold cracking, ultimately resulting in failure of the CVJ. If significant swelling or softening occurs, the maximum high speed capability of the boot is reduced due to the poor stability at speed and/or excessive radial expansion.

[0007] In order to solve the aforesaid problems, U.S. Pat. No. 6,656,890 B1 suggests a special base oil combination comprising 10 to 35% by weight of one or more poly-α-olefins, 3 to 15% by weight of one or more synthetic organic esters, 20 to 30% by weight of one or more naphthenic oils, the remainder of the combination being one or more paraffinic oils, and, further, a lithium soap thickener, and a sulphur-free friction modifier, that may be an organo-molybdenum complex, and molybdenum dithiophosphate, and a zinc dialkylthiophosphate and further additives such as corrosion inhibitors, anti-oxidants, extreme pressure additives, and tackiness agents. However, the friction coefficient and the wear of grease compositions according to U.S. Pat. No. 6,656,890 B1 as measured in SRV (abbreviation for the German words Schwingungen, Reibung, Verschleiß) tests needs to be improved.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] FIG. 1a is a graph of friction coefficient data for a grease embodiment of the present invention and several greases with commercial organic molybdenum containing additives;

[0009] FIG. 1b is a graph of wear data for a grease embodiment of the present invention and several greases with commercial organic molybdenum containing additives;

[0010] FIG. 2a is a graph of friction coefficient data for several grease composition embodiments of the present invention and several grease compositions with differing levels of tri-nuclear molybdenum compounds containing sulfur (TNMoS);

[0011] FIG. 2b graph of wear data for several grease composition embodiments of the present invention and several grease compositions with differing levels of tri-nuclear molybdenum compounds containing sulfur (TNMoS);

[0012] FIG. 3a is a graph of friction coefficient data for several grease composition embodiments of the present invention with varying levels of a zinc compound additive;

[0013] FIG. 3b is a graph of wear data for several grease embodiments of the present invention with varying levels of a zinc compound additive;

[0014] FIG. 4a is a graph of friction coefficient data for several different grease composition embodiments of the present invention with zinc compound additives and varying levels of an extreme pressure agent and varying levels of an additional sulfur containing compound;

[0015] FIG. 4b is a graph of wear data for several different grease composition embodiments of the present invention with zinc compound additives and varying levels of an extreme pressure agent and varying levels of an additional sulfur containing compound;

[0016] FIG. 5a is a graph of friction coefficient data for grease composition embodiments of the present invention and grease compositions lacking tri-nuclear molybdenum compounds containing sulfur (TNMoS) with varying amounts of thickeners; and

[0017] FIG. 5b is graph of wear data for grease composition embodiments of the present invention and grease composi-
tions lacking tri-nuclear molybdenum compounds containing sulfur (TNMoS) with varying amounts of thickeners.

**DETAILED DESCRIPTION**

**[0018]** Thus, it is the object of the present invention to provide for a grease composition, primarily for use in constant velocity joints, which has a good compatibility with boots made of rubber or thermoplastic elastomer, and which also gives low wear and low friction in use in CVJ.

**[0019]** Said object of the present invention is solved by a grease composition for use in constant velocity joints comprising

- (a) a base oil composition; and
- (b) 0.25% by weight to 5% by weight, preferably 0.3% by weight to 3% by weight, referred to the total amount of the grease composition, of at least one tri-nuclear molybdenum compound of the formula

\[ M_{1n}S_{1k}Q_0 \]

wherein \( L \) are independently selected ligands having organo groups with a sufficient number of carbon atoms to render the compound soluble or dispersible in the oil, \( n \) is from 1 to 4, \( k \) varies from 4 through 7, \( Q \) is selected from the group of neutral electron donating compounds such as amines, alcohols, phosphines, and ethers, and \( z \) ranges from 0 to 5 and includes non-stoichiometric values.

**[0020]** The number of carbon atoms present in the tri-nuclear molybdenum compound among all the ligands, organo groups is at least 21 carbon atoms, preferably at least 25, more preferably at least 30, and most preferably at least 35. Tri-nuclear molybdenum compounds usable in the present invention are disclosed in U.S. Pat. No. 6,172,013 B1, the disclosure of which is incorporated in the present invention insofar by reference. The inventors of the present invention have found that the presence of at least 0.25% by weight of the tri-nuclear molybdenum compound according to claim 1 would significantly lower the friction coefficient as well as the wear of CVJ in use. Surprisingly, the presence of 0.2% by weight or less of the tri-nuclear molybdenum compound would not lead to a much lowered friction coefficient nor to a lower wear when used in CVJ.

**[0021]** As a base oil composition according to the present invention, a base oil composition as disclosed in U.S. Pat. No. 6,656,890 B1, the disclosure of which is incorporated insofar herein by reference, may preferably be used. However, any further kind of base oil composition, especially a blend of mineral oils, a blend of synthetic oils or a blend of a mixture of mineral and synthetic oils may be used. The base oil composition should preferably have a kinematic viscosity of between about 32 and about 250 mm²/s at 40°C and between about and about 25 mm²/s at 100°C. The mineral oils preferably are selected from the group comprising at least one naphthenic oil and/or at least one paraffinic oil. The synthetic oils usable in the present invention are selected from a group comprising at least one poly-α-olefin (PAO) and/or at least one synthetic ester. The organic synthetic ester is preferably a di-carboxylic acid derivative having subgroups based on aliphatic alcohols. Preferably, the aliphatic alcohols have primary, straight or branched carbon chains with 2 to 20 carbon atoms. Preferably, the organic synthetic ester is selected from a group comprising sebacic acid-bis(2-ethylhexylester) ("dioctyl sebacate" (DOS)), adipic acid-bis(2-30 ethylhexylester) ("dioctyl adipate" (DOA)), and/or azelaic acid-bis(2-ethylhexylester) ("dioctyl azelate" (DOAZ)).

**[0025]** If poly-α-olefin is present in the base oil composition, preferably poly-α-olefins are selected having a viscosity in a range from about 2 to about 40 centistokes at 100°C. Naphthenic oils selected for the base oil compositions have preferably a viscosity in a range between 20 to 180 mm²/s at 40°C, whereas if paraffinic oils were present in the base oil composition, preferably the paraffinic oils have a viscosity in a range between about 25 to about 400 mm²/s at 40°C.

**[0026]** In a further embodiment of the present invention, the grease composition further comprises at least one zinc compound additive, more preferably a zinc compound additive in an amount of about 0.1% by weight to about 1.5% by weight. Most preferred the zinc compound additive is selected from the group comprising at least one of zinc dialkylphosphates (ZnDTP) and/or zinc dithiophosphates (ZnDTC), ZnO and/or ZnS. The zinc dialkylphosphat is preferably selected from the group of zinc dialkylphosphates of the following general formula:

\[ (R^1OR^2)OSP\quad S\quad Zn\quad S\quad P(OR^3)(OR^4) \]

wherein each of \( R^1 \) to \( R^4 \) inclusive may be the same or different and each represents a primary or secondary alkyl group having 1 to 24, preferably 3 to 20, most preferably 3 to 5 carbon atoms. In particular, excellent effects can be expected if the substituents \( R^1, R^2, R^3 \) and \( R^4 \) represent a combination of primary and secondary alkyl groups, each having 3 to 8 carbon atoms.

**[0027]** The zinc dithiophosphate may be preferably selected from zinc dialkylphosphates of the following general formula:

\[ R^4/\overset{\text{Zn}}{\text{C}}\text{S} \quad S \quad Zn \quad S \quad R^5 \]

wherein \( R^5, R^6, R^7, \) and \( R^8 \) may be the same or different and each represents an alkyl group having 1 to 24 carbon atoms or an aryl group having 6 to 30 carbon atoms.

**[0028]** By adding at least one zinc compound additive to the grease composition according to the invention, the friction coefficient as well as the wear in CVJ is diminished further significantly.

**[0029]** According to another embodiment of the invention, the grease composition further comprises a thickener selected from the group comprising lithium soaps, calcium soaps, lithium complex soaps, calcium complex soaps, and/or urea-derivative type thickener. The urea-derivative type thickener is not restricted to specific ones and maybe, for instance, also a diurea compound and/or a polyurea compound.

**[0030]** In the sense of the present invention, a lithium soap or a calcium soap is a reaction product of at least one fatty acid with lithium hydroxide or calcium hydroxide. Preferably, the thickener may be a simple lithium or calcium soap formed from stearic acid, 12-hydroxy stearic acid, hydrogenated castor oil or from other similar fatty acids or mixtures thereof or methylesters of such acids. Alternatively, a lithium and/or calcium complex soap may be used formed for example from a mixture of long-chain fatty acids together with a complexing agent, for example a borate of one or more dicarboxylic acids or a mixture of short and/or medium chain carboxylic acids. The use of complex lithium and/or calcium soaps allows the grease composition according to the present
invention to operate up to a temperature of about 180°C, whereas with simple lithium and/or calcium soaps, the grease composition will only operate up to a temperature of about 120°C. However, mixtures of all of the aforesaid thickeners may also be used.

[0031] According to a further embodiment of the present invention, the grease composition further comprises an additive package selected from the group of agents comprising antioxidation agents, corrosion inhibitors, anti-wear agents, friction modifiers, and/or extreme pressure agents (EP agents).

[0032] The EP agent is preferably a metal-free, sulphurised fatty acid methyl ester agent with a viscosity of about 25 mm²/s at 40°C being present preferably in an amount between about 0.1 to about 3% by weight, referred to the total amount of the grease composition. The total sulphur amount of the EP agent preferably ranges from about 8 to about 10% by weight and the active sulphur amount is about 1% by weight. Such EP agents exhibit excellent effects with respect to the prevention of seizure of CVJ. If the sulphur content exceeds the upper limit defined above, it may promote the initiation of rolling contact fatigue and wear of the contacting metal components.

[0033] As an anti-oxidation agent, the grease composition of the present invention may comprise an amine, preferably an aromatic amine, more preferably phenyl-naphthylamine or diphenylamine or derivatives thereof. The anti-oxidation agent is used to prevent deterioration of the grease composition associated with oxidation. The grease composition according to the present invention may range between about 0.1 to about 2% by weight, referred to the total amount of the grease composition, of an anti-oxidant agent in order to inhibit the oxidation degradation of the base oil, as well as to lengthen the life of the grease composition, thus prolonging the life of the CVJ.

[0034] Typically, the last operation before the assembly of CVJ is a wash to remove machining debris, and it is therefore necessary for the grease to absorb any traces of remaining water and to prevent the water from causing corrosion and adversely effecting the performance of the CVJ, it is therefore necessary to add a corrosion inhibitor. As a corrosion inhibitor, the grease composition according to the present invention may comprise at least one metal salt selected from the group consisting of metal salts of oxidised waxes, metal salts of petroleum sulphonates, especially prepared by sulphonating aromatic hydrocarbon components present in fractions of lubricating oils, and/or metal salts of alkyl aromatic sulphonates, such as dinonylnaphthalene sulphonic acids, alkylbenzenesulphonic acids, or overbased alkylbenzenesulphonic acids. Examples of the metal salts include sodium salts, potassium salts, calcium salts, magnesium salts, zinc salts, and quaternary ammonium salts, the calcium salts being most preferred. Calcium salts of oxidised waxes also ensure an excellent effect.

[0035] Anti-wear agents according to the present invention prevent a metal-to-metal contact by adding film-forming compounds to protect the surface either by physical absorption or chemical reaction. ZnDTP-compounds may also be used as anti-wear agents. As anticorrosion agents according to the present invention preferably calciumsulphonate salts are used, preferably an amount between about 0.5 to about 3% by weight, referred to the total amount of the grease composition.

[0036] Traditional friction modifiers such as fatty acid amides and fatty amine phosphates have been used in greases and other lubricants for many years (see, e.g., Klamann, Dieter —“Lubricants”, Verlag Chemie GmbH 1983, 1st edition, chapter 9.6). Their role is to give the lubricant stable but not necessarily low friction over a wide range of operating conditions.

[0037] In a further preferred embodiment of the present invention, a grease composition comprises about 55% by weight to about 97.5% by weight of the base oil composition, especially with a kinematic viscosity of between about 32 and about 250 mm²/s at 40°C. and between about 5 and about 25 mm²/s at 100°C, about 0.3% by weight to about 3% by weight of at least one tri-nuclear molybdium compound, about 0.1% by weight to about 1.5% by weight of at least one zinc compound additive and about 2% by weight to about 25% by weight of at least one thickener, in each case referred to the total amount of the grease composition. Preferably, an area thickener may be present in a range between about 5 to about 20% by weight, a lithium soap thickener between 2 to 15% by weight and a calcium complex soap thickener between about 2 to about 25% by weight.

[0038] Further, the grease composition according to the present invention has a sliding friction coefficient of not more than 0.1, as measured with a SRV test.

BEST MODE FOR CARRYING OUT THE INVENTION

[0039] In order to determine the effect of the lowering of the friction coefficient as well as the wear by the grease composition according to the invention, SRV tests are carried out using an Optimol Instruments SRV tester. Laser disc lower specimens made of the 100Cr6 standard bearing steel from Optimol Instruments Pruftechnik GmbH, Westendstrasse 125, Munich, properly cleaned using a solvent are prepared and contacted with the grease composition to be examined. The SRV test is an industry standard test and is especially relevant for the testing of greases for CVJs. The test consists of an upper ball specimen with a diameter of 10 mm made from a 100Cr6 bearing steel reciprocating under load on the flat disc lower specimen indicated above. In tests for mimicking tripod joints a frequency of 40 Hz with an applied load of 200 N were applied for 60 minutes (including running-in) at 80°C. The stroke was 1.5 mm and 3.0 mm, respectively. The friction coefficients obtained were recorded on computer. For each grease, the reported value is an average of four data at the end of tests in four runs (two runs at 1.5 mm stroke and two runs with 3.0 mm stroke). Wear is measured using a profilometer and a digital planimeter. By using the profilometer, a profile of the cross section in the middle of the worn surfaces can be obtained. The area (S) of this cross section can be measured by using the digital planimeter. The wear quantity is assessed by V=SI, where V is the volume of the wear and I is the stroke. The wear rate (W,) is obtained from W,=V/L [µm³/m], where L is the total sliding distance in the tests. For the running-in, it is started with an applied load of 50 N for 1 minute under the above-specified conditions. Afterwards, the applied load is increased for 30 seconds by 50 N up to 200 N.

[0040] The following substances are used in the examined grease compositions:

Base Oil Composition

[0041] The base oil compositions used have a kinematic viscosity of between about 32 and about 250 mm²/s at 40°C.
and between about 5 and about 25 mm²/s at about 40°C. Two base oil blends are used in this invention. The base oil blend A is a mixture of one or more naphthenic oils in a range between about 10 to about 60% by weight, one or more paraffinic oils in a range between about 30 to about 80% by weight and one or more polyalpha-olefins (PAO) in a range between about 5 to about 40% by weight, referred to the total amount of the oil mixture. Oil blend A does not contain an organic synthetic ester, whereas oil blend B contains DOS in a range between about 2 to about 10% by weight referred to a total amount of the oil mixture.

The naphthenic oils are selected with a range of viscosity between about 20 to about 180 mm²/s at 40°C, paraffinic oils between about 25 to about 400 mm²/s at 40°C, and PAO between about 6 and about 40 mm²/s at 100°C.

Tri-Molecular Molybdenum Compound (TNMoS)

The tri-molecular molybdenum compound used in the grease compositions according to the present invention is a sulphur-containing tri-nuclear molybdenum compound obtained under the trade name C9455B by Infineum International Ltd., USA. Its structure is defined in U.S. Pat. No. 6,172,013 B1.

Further Molybdenum Compounds for Comparative Examples

For comparative examples, a molybdenum dithiophosphate (MoDTP) sold under the commercial name RC3580 by Rhein Chemie Rheinau GmbH, Germany, with the chemical formula 2-Ethylhexyl molybdenum dithiophosphate, diluted with mineral oil, is used. Further, a molybdenum dithiocarbamate (MODTC) sold under the trade name Adeka Sulkuralube 600 (S-600) in the solid state and Sulkuralube 515 (S-515) in the liquid state, produced by Asahi Denka Co. Limited, Japan, is used. Further organo molybdenum complexes of organic amides (Organo Mo amide), sold under the trade name Molyvan 855 by R. T. Vanderbilt, USA, as well as one organo molybdenum complex of an amine (Organo Mo amine) sold under the trade name Salkuralube 700 (S-700), produced by Asahi Denka Co. Limited, Japan, are used.

Additives

As an anti-oxidant agent (Anti-oxidant), a diphenylamine with butyl and/or octyl groups is used, supplied by Ciba Specialty Chemicals, Switzerland under the trade name L-57 (Lignano L57). As an EP agent, a sulphurised organic compound (fatty acid methylester) sold under the trade name DeoAdd MD10 by DOG Deutsche Oelfabrik, Gesellschaft für chemische Erzeugnisse mbH und Co, Hamburg, Germany (“EP additive” in the examples), is used. Another example of an EP agent is a grease with calcium sulphonate thickeners, as produced by Brugarolas S.A., Spain, under the trade name Ca—S Grease (Ca—S grease).

As a corrosion inhibitor, a calcium salt of dinonylnaphthalene sulfonate, distributed for example by King Industries Co. Ltd., Norwalk, Conn., USA, under the trade name NaSul 72930 (Ca-sulphonate) is used.

First, the advantages of the grease composition according to the invention were examined by comparing the friction coefficient and wear of the same with other commercial organic molybdenum containing additives (example A). Six different grease compositions were produced, as listed in the following Table 1:

<table>
<thead>
<tr>
<th>Grease Composition</th>
<th>Example A1</th>
<th>Example A2</th>
<th>Example A3</th>
<th>Example A4</th>
<th>Example A5</th>
<th>Example A6</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNMoS</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>2.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>MoDTP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MoDTC (solid)</td>
<td></td>
<td></td>
<td>1.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Organo Mo amide</td>
<td></td>
<td></td>
<td></td>
<td>1.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Organo Mo amine</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>ZnDTP</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Anti-oxidant</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>oil blend A</td>
<td>81.75</td>
<td>81.75</td>
<td>81.75</td>
<td>80.75</td>
<td>81.75</td>
<td>82.75</td>
</tr>
<tr>
<td>Calcium complex soap</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
</tr>
</tbody>
</table>

Zinc Compound Additive

As zinc compound additives, ZnDTP, sold by Infineum International Ltd., UK, under the trade name Para-nox-15 or sold by Rhein Chemie, Germany, under the trade name RC3088 are used, being a zinc diallyldithiophosphate with primary and secondary alkyl groups, preferably diluted with mineral oil, is used. Further, ZnDTC sold under the trade name Vanlube A2 by R.T. Vanderbilt, USA, as well as ZnO and ZnS are used as zinc compound additives.

Thickener

As a lithium soap (Li soap), a reaction product of a fatty acid, such as stearic or 12-hydroxystearic with lithium hydroxide monohydrate is used. Further, a calcium complex soap (Calcium complex soap) being a reaction product of calcium hydroxide with two carboxylic acids, one with a short carbon chain length of 2 to 5 carbon atoms and one with a long carbon chain length of 16 to 20 carbon atoms, in which the short to long chain ratio is between 1:2 and 1:5 is used.

Furthermore, calcium stearate and a calcium stearate additive may be used as thickeners. Such additives are for instance Ca—S Grease (Ca—S grease).

Calcium complex soap

| Calcium complex soap | 16.0 | 16.0 | 16.0 | 16.0 | 16.0 | 16.0 |

Calcium complex soap, as such, is not used in the examples, but is included in the description of the thickeners.

[0067] The results from the SRV-measurement of the friction coefficient and the wear of examples A1 to A6 may be derived from FIG. 1. Only example A1 is a grease composition in accordance with the present invention, whereas examples A2 to A6 contain other commercial organic molybdenum-containing additives (A2 to A5) or no molybdenum containing additive (A6). The friction coefficient for example
A1 is clearly decreased when compared to the friction coefficient of the comparative examples, and is below 0.09. Further, the wear measured of example A1 is the lowest wear in the test series among examples A1 to A6, and is about 165 μm/m.

In a further series of tests, further grease compositions in accordance with the present invention were prepared containing different concentrations of the tri-nuclear molybdenum compound containing sulphur (TNMnMoS), as listed in Table 2.

<table>
<thead>
<tr>
<th>Grease Composition</th>
<th>Example B1</th>
<th>Example B2</th>
<th>Example B3</th>
<th>Example B4</th>
<th>Example B5</th>
<th>Example B6</th>
<th>Example B7</th>
<th>Example B8</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNMnMoS</td>
<td>0.0</td>
<td>0.1</td>
<td>0.2</td>
<td>0.3</td>
<td>0.5</td>
<td>1.0</td>
<td>2.0</td>
<td>3.0</td>
</tr>
<tr>
<td>ZnDTP</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Anti-oxidant</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>oil blend A</td>
<td>82.75</td>
<td>82.65</td>
<td>82.55</td>
<td>82.45</td>
<td>82.25</td>
<td>81.75</td>
<td>80.75</td>
<td>79.75</td>
</tr>
<tr>
<td>Calcium complex soap</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
</tr>
</tbody>
</table>

The results from SRV tests with respect to the friction coefficient and wear will be seen from FIG. 2.

As may be taken from FIG. 2a, surprisingly, the friction coefficient of grease compositions B1 to B3 is clearly about 0.1. Said grease compositions B1 to B3 are not in accordance with the present invention. One may easily see from FIG. 2a that concentrations of the sulphur containing tri-nuclear molybdenum compound of 0.2% by weight or less do not lower the friction coefficient significantly, whereas the grease composition B4 in accordance with the present invention shows a friction coefficient being lowered of at least about 25% when compared to examples B1 to B3. Thus, in accordance with the present invention only amounts of the sulphur containing tri-nuclear molybdenum compound of about 0.25% by weight, referred to the total amount of the grease composition, lead to an advantageous lowered friction coefficient and lower values for the wear, as will be seen from FIG. 2b.

In a third test series, the effect of the addition of a zinc compound additive to the grease composition according to the present invention was examined by preparing grease compositions in accordance with Table 3.

<table>
<thead>
<tr>
<th>Grease Composition</th>
<th>Example C1</th>
<th>Example C2</th>
<th>Example C3</th>
<th>Example C4</th>
<th>Example C5</th>
<th>Example C6</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNMnMoS</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>ZnDTP</td>
<td>0.5</td>
<td>1.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ZnDTC</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>ZnO</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.20</td>
</tr>
<tr>
<td>Anti-oxidant</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>oil blend A</td>
<td>81.75</td>
<td>81.25</td>
<td>80.75</td>
<td>80.75</td>
<td>81.55</td>
<td>81.55</td>
</tr>
<tr>
<td>Calcium complex soap</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
<td>16.0</td>
</tr>
</tbody>
</table>

The results from the SRV tests carried out with respect to examples C1 to C6 are shown in FIG. 3.

As will be seen when comparing example C1 having no zinc compound additive with examples C2 to C6, one will see that especially the wear is significantly lowered (not measurable) when adding a zinc compound additive to the grease composition according to the present invention. Further, also the friction coefficients are lowered and do not exceed the value of 0.08. Especially preferred is the addition of ZnDTP (C2 and C3) or the addition of ZnS (C6).
Further, the effect of adding an EP agent to the grease composition according to the present invention is demonstrated by preparing different grease compositions in accordance with Table 4.

<table>
<thead>
<tr>
<th>Grease Composition</th>
<th>Example D1</th>
<th>Example D2</th>
<th>Example D3</th>
<th>Example D4</th>
<th>Example D5</th>
<th>Example D6</th>
<th>Example D7</th>
<th>Example D8</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNMoS</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>ZnDTP</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>EP additive</td>
<td>0.1</td>
<td>0.3</td>
<td>0.3</td>
<td>0.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>Ca—S grease</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Anti-oxidant</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
<td>3.0</td>
</tr>
<tr>
<td>oil blend B</td>
<td>91.7</td>
<td>91.6</td>
<td>91.4</td>
<td>91.2</td>
<td>89.2</td>
<td>89.7</td>
<td>90.7</td>
<td>88.7</td>
</tr>
<tr>
<td>Li soap</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
</tr>
</tbody>
</table>

The results of the SRV tests of the friction coefficient and wear measurements will be seen from FIG. 5.

Finally, the effects of using different thickeners in the grease composition in accordance with the present invention is demonstrated by preparing different grease compositions in accordance with Table 5.

<table>
<thead>
<tr>
<th>Grease composition</th>
<th>Example E1 = A1</th>
<th>Example E2 = A6</th>
<th>Example E3 = D1</th>
<th>Example E4</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNMoS</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>ZnDTP</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Anti-oxidant</td>
<td>0.25</td>
<td>0.25</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>oil Blend A</td>
<td>81.75</td>
<td>80.75</td>
<td>91.7</td>
<td>92.7</td>
</tr>
<tr>
<td>oil Blend B</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calcium complex</td>
<td>16.0</td>
<td>16.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>soap</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Li soap</td>
<td>6.0</td>
<td>6.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Urea thickener</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

In summary, therefore, the grease composition according to the present invention has an advantageous significant influence on the friction coefficient and wear, leading to lower wear and lower friction in CVJ, and prevents the premature initiation of rolling contact fatigue in the joint.

1. A grease composition for use in constant velocity joints comprising:
   a) a base oil composition; and
   b) 0.25% by weight to 5% by weight of at least one trinuclear molybdenum compound of the formula
   \[ \text{MnS}_xL_nQ_3 \]
   wherein L are independently selected ligands having organo groups with a sufficient number of carbon atoms to render the compound soluble or dispersible in the oil, n is from 1 to 4, k varies from 4 though 7, Q is selected from the group of neutral electron donating compounds consisting of amines, alcohols, phosphines, ethers, and aze and ranges from 0 to 5 and includes non-stoichiometric values.

2. A grease composition according to claim 1, further comprising at least one zinc compound additive.

3. A grease composition according to claim 2, comprising at least one zinc compound additive in an amount of between 0.1% by weight to 2.5% by weight, referred to the total amount of the composition.

4. A grease composition according to claim 3 where the zinc compound is at least one selected from the group consisting of zinc dithiophosphates, zinc dithiocarbamates, zinc oxide, and zinc sulfide.

5. A grease composition according to claim 1, further comprising a thickener selected from the group consisting of lithium soaps, calcium soaps, lithium-complex soaps, calcium-complex soaps, urea-derivative type thickener, and mixtures thereof.

6. A grease composition according to claim 1, characterised in that the base oil composition comprises at least one of poly-[alpha]-olefins, naphthenic oils, paraffinic oils, and synthetic organic esters.

7. A grease composition according to claim 1, further comprising an additive package selected from the group of agents consisting of anti-oxidation agents, corrosion inhibitors, anti-
wear agents, friction modifiers, and/or extreme pressure agents, and mixtures thereof.

8. A grease composition according to claim 1, comprising 55% by weight to 97.5% by weight of the base oil composition, 0.3% by weight to 3% by weight of at least one tri-nuclear molybdenum compound, 0.1% by weight to 1.5% by weight of at least one zinc compound additive, and between 2% and 25% by weight of at least one thickener in each case referred to the total amount of the grease composition.

9. A grease composition according to claim 1, characterised in that the sliding friction coefficient of the grease composition is not more than 0.1.

* * * * *