LITHIUM COMPLEX GREASE WITH IMPROVED THICKENER YIELD

Provided is an improved lithium complex grease. The grease composition has a higher thickener yield, yet good mechanical properties. The grease composition comprises a lubricating oil, a lithium complex thickener and a copolymer, which in one embodiment may comprise styrene and butadiene.
LITHIUM COMPLEX GREASE WITH IMPROVED THICKENER YIELD

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a lithium complex grease having improved thickener yield. More particularly, a more economical yet effective lithium complex grease is provided having a higher weight of base oil incorporated into the grease matrix.

2. Description of the Related Art

Lithium complex greases have been known and manufactured for quite some time. Such greases can be made from any of a wide number of different base stocks of lubricating oil viscosity, and combination and mixtures of such stocks. The resulting greases are all marked by varying levels of desirable grease characteristics including dropping point, penetration, mechanical and/or shear stability, oxidation resistance, etc., all of which in combination are taken into account when describing the lubricating life of the grease.

Lubricating formulations and greases containing a wide assortment of different materials are described in the literature. Lithium complex greases comprising a lithium complex thickener and a lubricating base oil are well known.

There remains a need for greases, however, that keep penetration in an acceptable range while remaining economical and commercially acceptable.

SUMMARY OF THE INVENTION

Provided is a grease composition comprising a major amount of a base oil of lubricating viscosity, and a minor amount of a lithium complex thickener. The grease composition can further comprise a copolymer, for example, of styrene and butadiene. In general, the composition will comprise less than 25 wt% of the thickener in combination with the copolymer. The present grease composition exhibits a higher thickener yield, believed due to the combination of the lithium complex thickener and the copolymer, which in one embodiment is comprised of styrene and butadiene.

In one aspect of the present invention, a grease composition has been discovered which provides good mechanical properties, but also good economics. The thickener yield for the grease is much higher due to the presence of the copolymer of styrene and butadiene. The thickener yield can be at least 25% higher, 35% higher or even 50% higher than that of a composition without the copolymer.

TABLE I

<table>
<thead>
<tr>
<th>Group</th>
<th>Sulfur, ppm</th>
<th>Saturates, %</th>
<th>VI</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>&gt;300</td>
<td>And/or &lt;90</td>
<td>80-120</td>
</tr>
<tr>
<td>II</td>
<td>&lt;200</td>
<td>And ≥90</td>
<td>80-120</td>
</tr>
<tr>
<td>III</td>
<td>≥300</td>
<td>And ≥90</td>
<td>&gt;120</td>
</tr>
<tr>
<td>IV</td>
<td>All Polyalkylenes</td>
<td></td>
<td></td>
</tr>
<tr>
<td>V</td>
<td>All Stocks Not Included in Groups I-IV</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Facilities that make Group I lubricant base oils typically use solvents to extract the lower viscosity index (VI) components and increase the VI of the crude to the specifications desired. These solvents are typically phenol or furfural. Solvent extraction gives a product with less than 90% saturates and more than 300 ppm sulfur. The majority of the lubricant production in the world is in the Group I category.

Facilities that make Group II lubricant base oils typically employ hydroprocessing such as hydrotreating or severe hydrotreating to increase the VI of the crude oil to the specification value. The use of hydrotreating typically increases the saturate content above 90 and reduces the sulfur below 300 ppm. Approximately 10% of the lubricant base oil production in the world is in the Group II category, and about 30% of U.S. production is Group II.

Facilities that make Group III lubricant base oils typically employ wax isomerization technology to make very high VI products. Since the starting feed is waxy vacuum gas oil (VGO) or wax which contains all saturates and little sulfur, the Group III products have saturate contents above 90 and sulfur contents below 300 ppm. Fischer-Tropsch wax is an ideal feed for a wax isomerization process to make Group III lubricant base oils. Only a small fraction of the world’s lubricant supply is in the Group III category.

Group IV lubricant base oils are derived by oligomerization of normal alpha olefins and are called poly alpha olefin (PAO) lubricant base oils.

Group V lubricant base oils are all others. This group includes synthetic esters, silicon lubricants, halogenated lubricant base oils and lubricant base oils with VI values below 80. For purposes of this application, Group V lubricant base oils exclude synthetic esters and silicon lubricants. Group V lubricant base oils typically are prepared from petroleum by the same processes used to make Group I and II lubricant base oils, but under less severe conditions.

Synthetic lubricant base oils meet API Interchange Guidelines but are prepared by Fisher-Tropsch synthesis, ethylene oligomerization, normal alpha olefin oligomerization, or oligomerization of olefins boiling below C10.

The lithium complex thickener used in the present grease composition can be any known lithium complex thickener. For example, the lithium complex thickener can comprise a lithium soap derived from a fatty acid containing an epoxy group and/or ethylenic unsaturation and a dilithium salt derived from a straight chain dicarboxylic acid and/or, in one embodiment, a lithium salt derived from a hydroxy-substituted carboxylic acid such as salicylic acid.

The thickener can be a complex of a lithium soap of a C12 to C24 hydroxy fatty acid and a monolithium salt of boric acid and can include a lithium salt of a second hydroxy carboxylic acid such as salicylic acid.

The complex can comprise a lithium soap of a C12 to C24 hydroxy fatty acid thickener antioxidant comprising an
alkali metal salt of hydroxy benzoic acid and a diozime compound. The alkali metal salt of hydroxy benzoic acid include dilithium salicylate.

[0019] The complex can be a lithium soap which is a combination of a dilithium salt of a C₄ to C₁₂ dicarboxylic acid, e.g., dilithium azelate, a lithium soap of a 9-, 10- or 12-hydroxy C₃ to C₂₄ fatty acid, e.g., lithium 12-hydroxy stearate; and a lithium salt formed in-situ in the grease from a second hydroxy carboxylic acid wherein the —OH group is attached to a carbon atom not more than 6 carbons removed from the carboxyl group and wherein either of those groups may be attached to either aliphatic or aromatic portions of the materials.

[0020] Or, the lithium complex can comprise a combination of a complex lithium soap thickener, a lithium salt of a C₃ to C₁₂ hydroxy carboxylic acid and a thiadiazole. The grease may also optionally and preferably contain additional antioxidants, preferably amine type or phenol type anti-oxidants, most preferably amine type antioxidants.

[0021] In one embodiment, the lithium complex thickener is simply a lithium salt of a carboxylic acid, and in particular a hydroxy carboxylic acid, such as hydroxy stearic acid. Such a thickener can be prepared, for example, by reacting lithium hydroxyl monohydrate with the hydroxy stearic acid.

[0022] The greases will comprise a major amount, e.g., greater than 50% by weight of the base oil, and a minor amount of the thickener and any other additives, i.e., less than 50% by weight. The greases of the present invention may, of course, contain any of the other, typical grease additives such as rust inhibitors, barium dinonyl naphthalene sulfonate, oil modifiers, tackiness agents, extreme pressure agents, water shedding agents, dyes, etc. Typical additives and their function are described in Modern Lubricating Grease by C. J. Boer, Scientific Publication (G.B.) Ltd. 1976.

[0023] For the present invention, a copolymer is of particular significance as an additive. The copolymer can be a hydrocarbon based copolymer such as a copolymer of styrene and butadiene or ethylene and propylene. In one embodiment, the copolymer additive is a copolymer of styrene and butadiene. It has been found that use of a small amount of such a copolymer, e.g., from 2-6 weight percent, or from 2-5 weight percent, or in another embodiment, from 3-4 weight percent, in combination with a lithium complex thickener, results in a 25-50% increase in thickener yield.

[0024] Grease thickener yield, defined as the weight of base oil incorporated into the grease matrix per unit of weight of thickener, is a key determinant of mechanical grease properties, such as penetration, shear stability and water resistance as well as formula cost. The incorporation of the copolymer of styrene and butadiene with the lithium complex thickener permits the use of a greater amount of base oil, thereby raising the grease thickener yield, while maintaining good mechanical properties. The result is a more economical yet still effective grease.

[0025] Such copolymers of styrene and butadiene are commercially available. For example, V-211 is a copolymer of styrene and butadiene available from Functional Products, Inc. of Macedonia, Ohio. The copolymer is a white-yellow rubber powder which is quick dissolving. These copolymers of styrene and butadiene can be used in conjunction with other polymers, but they must be present in the grease compositions of the present invention.

[0026] By using the styrene/butadiene copolymer in combination with the lithium complex thickener, higher thickener yields are obtained. The yield, relating to the amount of base oil one incorporates into the grease matrix while still realizing good mechanical properties, can increase by at least about 25 weight percent, about 35 weight percent, or at least about 50 weight percent. Such grease compositions have been shown to exhibit worked penetration properties of at least about 290, at least about 300, and even at about 310. The compositions can be used in any automotive grease or industrial grease application. For example, the grease compositions can be used for wheel bearings, an automotive chassis or for lubricating gears.

EXAMPLES

[0028] Several greases were prepared as follows:

[0029] 1-50 wt % of formula amount of base oil(s) was mixed in an open kettle at room temperature. Formula amount of mono and di-acids, emulsifier(s) are added with water [at 50% base oil amount] and stirred while heating to 200°F. At 200°F, lithium hydroxide is added slowly. Once the lithium hydroxide had been added, the mixture was heated to 400°F. It was held at 400°F while stirring for 20 minutes. The grease is then cooled to 175°F while stirring. At 175°F, performance additives e.g., rust inhibitor, extreme-pressure and anti-wear agents and polymers, tackifiers are added. The grease penetration was adjusted by adding base oil slowly while stirring. When penetration range was achieved, the grease was cooled to room temperature and stored suitably.

[0030] Penetration of the greases was tested in accordance with ASTM 217. The results are shown in the Table below. In the Table, the various additives used are as follows:

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>PENNZOIL 600 HC</td>
<td>57.31</td>
<td>57.31</td>
<td>57.31</td>
<td>57.31</td>
<td>57.31</td>
<td>57.31</td>
<td>57.31</td>
<td>57.31</td>
</tr>
</tbody>
</table>

[0031] 1GEPAL CO-430. Sulfonic N-40, Naphthenic Acid: emulsifiers

[0032] Lubrizol 528C, EC5426A: rust inhibitor

[0033] glycerin, Lubrizol 5200: extreme-pressure, anti-wear agents

[0034] Lubrizol 2017: PIB polymer

[0035] Lubrizol 2002D: functionalized olefin polymer

[0036] V-211: copolymer of styrene-butadiene

[0037] V-207: copolymer of ethylene and propylene
Base Oil Content, wt %

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
</tr>
</thead>
</table>

Penetration: Worked P(60)

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.93</td>
<td>2.93</td>
<td>2.93</td>
<td>2.93</td>
<td>2.93</td>
<td>2.93</td>
<td>2.93</td>
<td>2.93</td>
</tr>
</tbody>
</table>

ASTM 294 319 D217

C D 10.26 10.26 3.14 3.14 2.93 2.93 0.5 0.5

LUBRIZOL 5283C

EC5425A

Glyceryl

LUBRIZOL 5200

LUBRIZOL 2017

LUBRIZOL 2002D

POLYMERS

V211

V207

[0038] Runs A and B are considered as base cases using a PIB polymer or a functionalized olefin polymer. In both cases, the base oil amount used is 80% of formula amount. In runs C and D, when these polymers are replaced with styrene-butadiene or ethylene-propylene copolymers, the used base oil amount increases to 157 and 132% of formula amount respectively. With an increasing amount of base oil in the formula, thickener yield increases accordingly. Runs E, F, G, and H show increased thickener yield with styrene-butadiene copolymer use in conjunction with PIB and functionalized polymers.

[0039] Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention. Other objects and advantages will become apparent to those skilled in the art from a review of the preceding description.

That which is claimed is:

1. A grease composition having improved thickener yield, comprised of a major amount of a lubricating base oil, and a minor amount of a lithium complex thickener.

2. The grease composition of claim 1, where the amount of thickener and any additives is less than 25 wt % of the grease composition.

3. The grease composition of claim 1, further comprising a copolymer of styrene and butadiene.

4. The grease composition of claim 3, wherein the copolymer is an ethylene-propylene copolymer.

5. The grease composition of claim 3, wherein the thickener yield is at least 25% higher than that of a composition with no copolymer.

6. The grease composition of claim 3, wherein the thickener yield is at least 50% higher than that of a composition with no copolymer.

7. The grease composition of claim 3, further comprising a copolymer of ethylene and propylene.

8. The grease composition of claim 1, wherein the lithium complex thickener comprises a lithium salt of a carboxylic acid.

9. The grease composition of claim 8, wherein the lithium complex thickener comprises a lithium salt of stearic acid.

10. The grease composition of claim 8, wherein the lithium complex thickener comprises a reaction product of lithium hydroxyl monohydrate and hydroxyoleic acid.

11. The grease composition of claim 8, wherein the amount of styrene-butadiene copolymer ranges from about 2-6 weight percent.

12. The grease composition of claim 11, wherein the amount of styrene-butadiene copolymer ranges from about 3-4 weight percent.

13. The grease composition of claim 1, wherein the composition has a worked penetration of at least about 290.

14. The grease composition of claim 1, wherein the composition has a worked penetration of at least about 300.

15. The grease composition of claim 3, wherein the composition has a worked penetration of at least about 310.

16. The grease composition of claim 6, wherein the lithium complex thickener comprises a lithium salt of a carboxylic acid, the amount of styrene-butadiene copolymer ranges from about 2-6 weight percent, and the composition has a worked penetration of at least about 300.

17. A method of lubricating wheel bearings, which comprises coating the wheel bearings with the grease composition of claim 1.

18. A method of lubricating wheel bearings, which comprises coating the wheel bearings with the grease composition of claim 16.

19. A method of lubricating an automotive chassis, which comprises using the grease composition of claim 3 to grease the chassis.

20. A method of lubricating gears, which comprises applying the grease composition of claim 3 to the gears.

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