United States Patent Office 3,472,770
Patented Oct. 14, 1969

3,472,770

NOVEL PINION GREASE

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No Drawing. Continuation-in-part of application Ser. No. 449,610, Apr. 20, 1965. This application Sept. 8, 1967, Ser. No. 666,482
Int. Cl. C10m 7/22, 7/02
U.S. Cl. 252—23 10 Claims

ABSTRACT OF THE DISCLOSURE

Greases, particularly useful for open gears, are provided combining polyisobutylene and an oxidized bright stock in combination with a complex calcium grease and solid lubricant fillers selected from molybdenum disulfide, graphite, and carbon black.

CROSS-REFERENCES TO RELATED APPLICATIONS

This application is a continuation-in-part of application Ser. No. 449,610, filed Apr. 20, 1965, now abandoned.

BACKGROUND OF THE INVENTION

Field of the invention

Open gears or pinion gears require lubricants having properties over and above those properties required for lubricants of moving parts where the lubricant is encased. A satisfactory pinion gear lubricant must have good adhesive and cohesive properties, must be easy to apply, must have a good penetration/temperature relationship and should provide a durable film between surfaces.

The adhesive and cohesive properties are balanced so that the lubricant will adhere to the metal for protection and lubrication purposes and at the same time be cohesive enough to feed well. A product which is too cohesive will "ball up" and will not adhere to a metal surface; one which is too adhesive will be difficult to apply. Moreover, a premium pinion grease should be sufficiently "grease-like" for it to be applied easily. The product should be dispensable from a tube when used in a heavy duty ratchet-type grease gun (calking gun) and should also be easy to apply with a paddle from a pail or drum.

Description of the prior art

Prior art pinion greases have relied in many instances on an inorganic thickener such as clay. The prior art has been aware of the use of polyisobutylene for its adhesive properties. See for example U.S. Patents Nos. 2,062,346 and 2,607,732. While oxidized bright stock is a well-known material, the combination of oxidized bright stock and polyisobutylene is novel. Moreover, the combination of polyisobutylene and oxidized bright stock and a calcium complex grease is heretofore unknown.

SUMMARY OF THE INVENTION

Pinion greases are provided by combining a relatively low molecular weight polyisobutylene with a high viscosity oxidized bright stock as a base material, and incorporating a calcium complex grease and preferably a carbon black filler. In the combination of polyisobutylene and oxidized bright stock, there should not be less than about 15 percent by weight of either material. The final product, containing the above combination plus the complex calcium grease, is found to have an excellent temperature/penetration relationship over a wide range so that it does not unreasonably soften at elevated temperatures nor become too viscous to flow at relatively low temperatures.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The pinion greases of this invention have four components: polyisobutylene; oxidized bright stock; calcium complex grease; and fillers. The following table indicates the relative proportions of the various materials.

Compound	Weight percent of total composition preferred	
Calcium complex grease (including oil)	10–40	15–35
Polyisobutylene	15–50	20–45
Oxidized bright stock	8–50	10–45
Fillers	3–25	5–20

The weight ratio of polyisobutylene to oxidized bright stock will generally be in the range of 1:0.25–5.

Small amounts of other additives may also be present in the grease. These include such additives as antioxidants, rust inhibitors, and stringiness agents. Illustrative of such additives are diphenyl amine, sodium nitrite, rubber latex, etc. Generally, these specialized types of additives will be present in total amount of about 0.1 to 4 weight percent of the total grease composition, more usually from about 0.1 to 2 weight percent of the total composition.

The grease is prepared by combining the various ingredients by any conventional mechanical means. The order of addition is not critical. Preferably, temperatures above 180° F. are used, usually from about 180° F. to 220° F., in order to facilitate mixing. Ordinarily, the fillers are usually added after the other ingredients have been combined.

The various ingredients will now be considered individually.

BRIGHT STOCK

Bright stock is a heavy distillate fraction usually obtained as a residue after the light fractions have been removed and which has been further purified to remove polynuclear aromatics, sulfur and nitrogenous-containing materials. Various extraction procedures have been used for the removal of the above materials, the Duo-sol process being among the more common. See, for example, Kalichevsky et al., Chemical Refining of Petroleum, Reinhold Publishing Corporation, 1942, p. 337.

The bright stcok will generally have a viscosity in the range of about 125 to 230 Saybolt Universal seconds (SUS) at 210° F. The bright stocks have high flash points, above 600° F., because they are normally the bottoms obtained from a distillation column.

The bright stock can be oxidized to oxygenated materials by blowing air through the bright stock at elevated temperatures and generally in the presence of an oxidation catalyst, e.g., iron or cobalt salts. The bright stock used in this invention is oxidized by various means known in the art to a Saybolt viscosity at 210° F. SUS (V_{210}) in the range of 10,000 to 30,000, preferably in the range of 14,000 to 18,000.

POLYISOBUTYLENE

The polyisobutylene which finds use has a V_{210} SUS in the range of 1,000 to 6,000, preferably 2,000 to 4,000. The polyisobutylene may be prepared by various means known in the art. The method of preparation of polyisobutylene is not critical to this invention, and any means which achieves the desired degree of polymerization or viscosity is satisfactory.

CALCIUM COMPLEX GREASE

The calcium complex grease thickeners used in this invention are a combination of a long-chain fatty acid calcium salt and calcium carbonate and/or a calcium salt of a low molecular weight fatty acid. Illustrative of cal-

cium complex greases is a grease composition described in U.S. Patent No. 3,186,944. Other calcium complex greases have been reported in U.S. Patents Nos. 3,068,175, 3,071,547, 3,077,450, 3,152,079, and 3,155,619. This list is not intended to be exhaustive but merely illustrative of various calcium complex greases employing calcium acetate and/or calcium carbonate and the calcium salt of a higher fatty acid.

Generally, the calcium complex greases are prepared having from about 10 to 30 weight percent, more usually from about 15 to 25 weight percent of solids (e.g., calcium salts). The remaining material will be for the most part a lubricating mineral oil having a Saybolt viscosity at 100° F. (V_{100}) in the range of from about 100 to 2,000 SUS.

The mol ratio of calcium acetate to the long-chain fatty acid (10 to 24 carbon atoms, e.g., calcium 12-hydroxystearate or calcium stearate) in the grease will generally be in the range of about 2–5:1. The calcium carbonate, when present, will generally be in the range of about 1 to 6 weight percent of the total grease composition.

While not essential, dispersants may be used with the complex calcium greases, particularly those greases which employ a combination of calcium hydroxystearate, calcium acetate and calcium carbonate. (See U.S. Patent No. 3,186,944.) These polymeric dispersants include copolymers of methacrylate esters and vinyl pyrrolidone, polyglycol polymers, as well as non-polymeric quaternary ammonium nitrites and quaternary ammonium chlorides. These particular dispersants are found in U.S. Patents Nos. 3,159,575, 3,159,576, and 3,189,543. Generally, the amount of the dispersant will be from about 0.1 to 0.5 weight percent of the complex calcium grease composition.

Illustrative dispersants are cocotrimethyl ammonium chloride, polyethylene glycol-methacrylate polymers, dodecyl methacrylate-vinyl pyrrolidone copolymers, etc.

FILLERS

The fillers are, for the most part, molybdenum disulfide and carbon, e.g., graphite or carbon black. The molybdenum disulfide will generally be present in from about 1 to 10 weight percent of the total pinion grease composition.

A carbon filler will usually be present. The carbon fillers include graphite, carbon blacks and lamp blacks and their chemical and physical equivalents. The carbon filler will vary, for the most part, in particle size and structure, absorption properties and tinting strengths. Wide variations in size and physical properties can be tolerated. Carbon fillers generally range in size from about 50 to 3000 A., preferably 100 to1000 A. Of particular use are the channel blacks and graphite.

The total amount of carbon filler usually will be at least one percent of the total composition and generally not exceed 15 percent. Preferably, a mixture of graphite and carbon black is used wherein at least one weight percent of the total composition of each of the carbon fillers is used.

The carbon black will usually be present in from 0 to 15 weight percent of the total composition, while graphite will generally be present in from 0 to 5 weight percent of the total composition.

Additional fillers may also be included, such as zinc oxide and lead oxides.

PROPERTIES OF THE GREASE

The ASTM worked penetration at 77° F. (60 strokes) will be at least 300 and usually at least 325. The dropping point will be in excess of 580° F. The greases have high load failures in the four-ball weld test and also provide good rust protection. The penetration/temperature relationship is found to provide a relatively flat curve so that softening and hardening are not excessive over a wide temperature range.

EXAMPLES

The following examples are offered by way of illustration and not by way of limitation.

Example A.—Preparation of calcium complex grease

A mixture of 7.9% of methyl 12-hydroxystearate and 45% of a California solvent-refined paraffinic base oil having a viscosity of 480 SUS at 100° F. was blended at room temperature, then heated to a temperature ranging from 180° F. to 190° F. The mixture was maintained at this temperature for a time sufficient to disperse the methyl 12-hydroxystearate in the oil. The mixture was then cooled to 130° F. followed by adidtion of 15 percent by weight of calcium hydroxide, after which the mixture was agitated at 130° F. to obtain a uniform dispersion in the oil. There was then added 0.1 weight percent sodium hydroxide as a 1 weight percent aqueous solution, and the whole mixture heated to 180°–200° F. for a period of 30 minutes, at which time 8.0 weight percent of glacial acetic acid was added over a period of time of about 30–45 minutes. Urea (2.2 weight percent) was then added, followed by sealing the mixture from the atmosphere and raising the temperature to 320° F. at 70–90 p.s.i. for a period of about 1 hour. The mixture was then vented to the atmosphere and heated at 330° F. for a period of one hour at atmospheric pressure. The balance of the oil was then added to make a total of 100%.

Example B.—Oxidation of bright stock

Bright stock (V_{210}=230 SUS) was mixed with a small amount of ferric chloride and heated to 425° F., while blowing air through the mixture until the desired viscosity was obtained. At this time, the mixture was cooled and the product isolated.

Example I

Into a heavy-duty grease mixer was added 42 parts of oxidized bright stock as described in Example B (Saybolt viscosity of 210° F.=15,000 SUS: ASTM penetration unworked at 77° F.=215), 22 parts of polyisobutylene having a Saybolt viscosity at 210° F. in the range of 2800–3200 SUS, 5 parts of molybdenum disulfide, 1 part of carbon black in oil and 30 parts of a calcium complex grease prepared as described in Example A. The mixture was then cooled to 180° F. and packaged.

The ASTM unworked penetration was 265, the worked penetration was 360, the ASTM dropping point was 580+° F.

When applied to open gears, the grease was found to provide excellent protection, even at elevated temperatures above 200° F.

Example II

Into a grease mixer was introduced 865 lbs. of calcium complex grease prepared as described in Example A and 3,424 lbs. of polyisobutylene having a Saybolt viscosity of 210° F. in the range of 2800–3200 SUS. Heating was begun, and 1,040 lbs. of carbon black (Cabot Elf-5, supplied by the Cabot Corporation, Boston, Mass.) in a polyethylene bag was added to the mixture with stirring and the temperature raised to 340° F. (The polyethylene becomes incorporated in the mixture.) When the polyethylene was well dispersed, the reaction mixture was cooled by adding 160 lbs. of powdered graphite, 120 lbs. molybdenum disulfide, 800 lbs. of oxidized bright stock prepared as described in Example B (Saybolt viscosity at 210° F.=15,000 SUS; ASTM penetration unworked at 77° F.=215), 1,295 lbs. of the calcium complex grease prepared as described in Example A, 240 lbs. of calcium sulfonate having 1.67 weight percent calcium derived by the sulfonation of 480 neutral oil and neutralization and 156 lbs. of tetraethylene glycol di(alkylphenol) ether (alkyl of about 14 carbon atoms) while the temperature is maintained at 200° F. The mixture is then cooled to 180° F.–190° F. and packaged. The ASTM worked penetration is 348.

The following tests were carried out to demonstrate the excellent properties of the pinion greases of this invention.

When applied to open gears, the greases were found to provide excellent protection, even at elevated temperatures above 200° F.

The penetration/temperature relationship determined as percent change for the grease of Example I was found to be 18% in going from 77° F. to 36° F. and 16% in going from 77° F. to 130° F., while the grease of Example II at the former temperature range was 14% and at the latter temperature range was 15%.

The greases were found to be readily fed from a calking gun at 36° F.

Using a modified Timken retention test, 25 pounds at 30 minutes, the greases were found to pass. See, "Manufacture and Application of Lubricating Greases," Boner, Reinhold Publishing Corporation, New York, 1954, pp. 867 ff. for a description of this and the following test. Using the four-ball weld point/extreme pressure test, the results for Grease I were pass—370 kg. load/fail—380 kg. load; for Grease II, pass—285 kg. load/fail—300 kg. load.

The pinion greases were also found to pass the humidity cabinet rust test for three days. Sandblasted steel panels ⅛" x 2" x 4 were dipped in the test greases and then placed in the humidity cabinet at 120° F. and 100% humidity.

It is evident that the greases of this invention provide excellent protection to open gears, fulfilling the many requirements necessary for an acceptable pinion grease.

As will be evident to those skilled in the art, various modifications on this invention can be made or followed, in the light of the foregoing disclosure and discussion, without departing from the spirit or scope of the disclosure or from the scope of the following claims.

What is claimed is:

1. Pinion grease having a relatively flat penetration/temperature profile comprising from 15 to 50 weight percent of polyisobutylene having a Saybolt viscosity at 210° F. in the range of 1000 to 6000 SUS, from 8 to 50 weight percent of oxidized bright stock having a Saybolt viscosity at 210° F. in the range of 10,000 to 30,000 SUS, the weight ratio of polyisobutylene to oxidized bright stock being in the range of 1:0.25–5 from 10 to 40 weight percent of a calcium complex grease and from 3 to 25 weight percent of solid lubricant filler.

2. A pinion grease according to claim **1** wherein said filler are comprised of at least one of molybdenum disulfide graphite or carbon black.

3. A pinion grease according to claim **1** wherein said polyisobutylene is present in from 20 to 45 weight percent.

4. A pinion grease according to claim **1** wherein said oxidized bright stock has a Saybolt viscosity at 210° F. in the range of 14,000 to 18,000 SUS and said polyisobutylene has a Saybolt viscosity at 210° F. in the range of 2000 to 4000 SUS.

5. A pinion grease according to claim **1** wherein said calcium complex grease is present in from 15 to 35 weight percent and contains calcium hydroxystearate, calcium acetate, and calcium carbonate.

6. A pinion grease according to claim **1** having from 1 to 15 weight percent of channel black.

7. A pinion grease according to claim **1** having from 1 to 10 weight percent of molybdenum disulfide, from 1 to 15 weight percent of a carbon filler and a calcium complex grease is from 15 to 35 weight percent containing calcium hydroxystearate, calcium acetate, and calcium carbonate.

8. A method of lubricating open gears avoiding throwoff of the lubricant and providing adhesive lubricating film which comprises applying to the open gears in an amount sufficient to provide lubrication a composition according to claim **1**.

9. A method of lubricating open gears avoiding throwoff of the lubricant and providing adhesive lubricating film which comprises applying to the open gears in an amount sufficient to provide lubrication a composition according to claim **5**.

10. A method of lubricating open gears avoiding throwoff of the lubricant and providing adhesive lubricating film which comprises applying to the open gears in an amount sufficient to provide lubrication a composition according to claim **7**.

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U.S. Cl. X.R.

252—18, 22, 55