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54 **A lubricating oil composition.**

57 The lubricating oil composition comprises 15-85 parts by weight of a poly- α -olefin having a kinematic viscosity of 1.5-150 centistokes at 100°C and 85-15 parts by weight of a mineral oil having a kinetic viscosity of 2-50 centistokes at 100°C and a pour point of -35°C or below. By virtue of the admixture of the mineral oil, the lubricant oil composition is much more inexpensive than the poly- α -olefin per se while retaining the excellent properties inherent in the poly- α -olefins such as the stability against oxidation, low-temperature performance, high-temperature cleanness and friction characteristics with wet clutches.

A LUBRICATING OIL COMPOSITION

BACKGROUND OF THE INVENTION

The present invention relates to a lubricating oil composition or, more particularly, to a lubricating oil composition having excellent stability against oxidation, performance at extremely low temperatures, cleanness at high temperatures and friction characteristics in wet clutches and usable in a wide variety of applications, for example, as an engine oil in internal combustion engines, ATF oil, lubricating oil in wet clutches for agricultural tractors and the like, compressor oil, gear oil, bearing oil and so on.

As is known, poly- α -olefins as a class of synthetic lubricating oils are utilized as a base oil of high-grade lubricating oils by virtue of their excellent stability against oxidation and fluidity at low temperatures. In addition to the economical disadvantage due to the expensiveness, however, poly- α -olefins have some technical problems when they are used, for example, as an engine oil in internal combustion engines or lubricating oil in wet clutches including poor cleanness at high temperatures, insufficient friction characteristics with wet clutches and others.

With an object to solve the economical problem, on the other hand, it is proposed to admix the poly- α -olefin with a mineral oil. This measure, however, never provides a true technical solution of the problem since admixture of a mineral oil may badly affect the excellent performance at extremely low temperatures and stability against oxidation inherent in the poly- α -olefins.

SUMMARY OF THE INVENTION

Thus, the present invention has an object to provide a novel poly- α -olefin based lubricating oil composition of high economical feasibility having excellent cleanness at high temperatures and friction characteristics with wet clutches without being affected in respect of the stability against oxidation and performance at extremely low temperatures. The extensive investigations undertaken with this object have unexpectedly led to a discovery that the object can be fully achieved by blending a poly- α -olefin with a mineral oil only when a poly- α -olefin having specific properties is combined with a mineral oil having specific properties in a specific proportion.

The lubricating oil composition of the invention formulated on the base of the above mentioned discovery comprises:

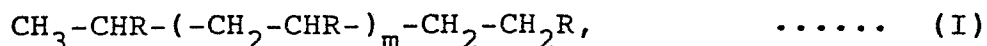
(A) from 15 to 85 parts by weight of a poly- α -olefin having a kinematic viscosity in the range from 1.5 to 150 centistokes at 100 °C; and

(B) from 85 to 15 parts by weight of a mineral oil having a kinematic viscosity in the range from 2 to 50 centistokes at 100 °C and a pour point of -35 °C or below.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

As is understood from the above given summary of the invention, the inventive lubricating oil composition is a mixture of a specific poly- α -olefin as the component (A) and a specific mineral oil as the component (B) in a specific weight proportion. The poly- α -olefin as the component (A) is a type of synthetic lubricating oil also called an α -olefin oligomer and represented by the general

formula



in which R is an alkyl group having 4 to 12 carbon atoms and the subscript m is zero or a positive integer not exceeding 30. The poly- α -olefin should have a kinematic viscosity in the range from 1.5 to 150 centistokes or, preferably, from 2 to 50 centistokes at 100 °C. The poly- α -olefin should have a relatively low degree of polymerization and particular examples of preferable poly- α -olefins include dimers to decamers of 1-octene, 1-decene or 1-dodecene, of which dimer, trimer and tetramer of 1-decene are more preferable.

Such a poly- α -olefin can be prepared by a known method including the steps of polymerization, decomposition of the catalyst, distillation and hydrogenation. A means to control the degree of polymerization of the poly- α -olefin is to control the staying time of the reactants in the reaction vessel in the step of polymerization.

The mineral oil as the component (B) to be combined with the above described poly- α -olefin as the component (A) should have a kinematic viscosity in the range from 2 to 50 centistokes or, preferably, from 5 to 35 centistokes at 100 °C and a pour point of -35 °C or below or, preferably, -40 °C or below. The content of aromatic hydrocarbons in the mineral oil, referred to as % C_A hereinbelow, should be 20% or lower or, preferably, 10% or lower. The content of sulfur therein should be 50 ppm or lower or, preferably, 5 ppm or lower. Use of a mineral oil containing more than 50 ppm of sulfur is undesirable due to the decreased stability of the resultant lubricating oil composition against oxidation.

Mineral oils satisfying such requirements can be obtained from a paraffinic crude oil or an intermediate base crude oil by distilling the same to give a distillate having a boiling point of 250 to 450°C under normal pressure which is then refined by a conventional method followed by a deep dewaxing treatment.

The distillate here implied is obtained by subjecting a crude oil to distillation under normal pressure or by subjecting a residue oil from distillation under normal pressure to distillation under reduced pressure. Though not particularly limitative, the distillate can be refined by one of the following five methods including: (1) hydrogenation treatment of the distillate, optionally, followed by alkali distillation or scrubbing with sulfuric acid; (2) solvent treatment of the distillate, optionally, followed by alkali distillation or scrubbing with sulfuric acid; (3) hydrogenation treatment of the distillate in two steps; (4) hydrogenation treatment of the distillate in three steps; and (5) hydrogenation treatment of the distillate in two steps followed by alkali distillation or scrubbing with sulfuric acid. Following description is exemplary of a process for the treatment of the distillate.

A paraffinic crude oil or an intermediate base crude oil is processed according to a conventional procedure to give a base material for lubricating oil which is then subjected to an extensive hydrogenation treatment. By this treatment, certain constituents in the base material undesirable for the fraction of lubricating oils, such as the aromatic matter, can be removed or converted into effective constituents along with removal of the sulfurous and nitrogenous matters almost to completeness.

The base material after the hydrogenation treatment is then

subjected to fractional distillation under reduced pressure so as to be imparted with a necessary viscosity. Thereafter, the oil is subjected to a dewaxing treatment using a solvent according to a known procedure to such an extent that the oil should have a pour point, which conventional paraffin-based oils may have, in the range, for example, from -15°C to -10°C .

If necessary, this dewaxing treatment is followed by a further treatment of hydrogenation to such an extent that the aromatic compounds contained in the base oil are mostly hydrogenated and converted into saturated compounds to impart the base oil with increased thermal and chemical stability. The thus refined base oil is still not suitable as a component of the inventive lubricating oil composition due to the high pour point. Accordingly, the above described refining treatment should be followed by a deep dewaxing treatment. This dewaxing treatment can be performed either by the method of solvent dewaxing under extensive conditions or by the method of catalytic hydrogenation dewaxing using a zeolite as the catalyst, in which the paraffin compounds or, mainly, normal paraffins adsorbed in the pores of the catalyst are preferentially decomposed under the atmosphere of hydrogen so that the base oil is freed from the constituents responsible for the formation of waxy materials.

Although the conditions of the hydrogenation treatment depend on the properties of the base oil and other factors, the process parameters usually include a reaction temperature in the range from 200 to 480°C or, preferably, from 250 to 480°C , pressure of hydrogen in the range from 5 to 300 kg/cm^2 or, preferably, from 30 to 250 kg/cm^2 and volume of the hydrogen gas introduced in the

range from 30 to 3000 Nm³ or, preferably, from 100 to 2000 Nm³ per kiloliter of the feed of the distillate. The catalyst suitable for this catalytic hydrogenation treatment can be prepared by using alumina, silica, silica-alumina, zeolite, active carbon, bauxite and the like as a carrier on which a metallic element belonging, for example, to the VIth or VIIIth Group of the Periodic Table or, preferably, cobalt, nickel, molybdenum, tungsten and the like is supported as the catalytically effective ingredient according to a known method for catalyst preparation. The catalyst should preferably be subjected to a preliminary sulfurization treatment prior to use.

As is mentioned above, the distillate oil after the hydrogenation treatment is subjected to one or more of different post-treatments. When the post-treatment includes the second-step or, further, third-step hydrogenation treatment, in particular, the process parameters in these subsequent hydrogenation treatments can be selected within the above described ranges and the conditions in each of the first-step to third-step hydrogenation treatments can be the same as or different from those in the other steps. It is usually preferable, however, that the hydrogenation in the second step and in the third step should be performed more extensively than in the first step and in the second step, respectively.

The alkali distillation to follow is undertaken with an object to remove any trace amount of the acidic substances contained in the hydrogenated oil so as to improve the stability thereof. This process is performed by the distillation

of the distillate oil with admixture of an alkali such as sodium hydroxide, potassium hydroxide and the like under reduced pressure.

The scrubbing of the oil with sulfuric acid, which is alternative to the alkali distillation, is a treatment conventionally performed as a finishing step of various petroleum products and undertaken here with an object to improve the properties of the distillate oil by removing aromatic hydrocarbons or, in particular, polycyclic aromatic hydrocarbons, olefins, sulfur compounds and so on therefrom. In the preparation of the mineral oil for the inventive lubricating oil composition, the distillate oil after the hydrogenation treatment is contacted with concentrated sulfuric acid in an amount of 0.5 to 5% by weight at a temperature in the range from room temperature to 60°C followed by neutralization with an alkali such as sodium hydroxide.

As is described in the above by (1) to (5), the distillate oil is treated in one of the combinations of the above described unit procedures while the procedures of (1), (3) and (4) are particularly preferable.

The distillate oil after the treatment in the above described manner has a kinematic viscosity of 2 to 50 centistokes at 100°C and a pour point of -35°C or below and the content of the aromatic hydrocarbons % C_A therein does not exceed 20%.

The lubricating oil composition of the present invention is composed of the above described poly- α -olefin and the

mineral oil. The composition should be composed of from 15 to 85% by weight or, preferably, from 20 to 80% by weight of the former component and from 85 to 15% by weight or, preferably, from 80 to 20% by weight of the latter component. The above given range is critical in order that the inventive composition may have excellent cleanness at high temperatures and friction characteristics with a wet clutch.

Although the essential ingredients in the inventive lubricating oil composition are the above described poly- α -olefin and mineral oil, it is optional that the lubricating oil composition is admixed with various kinds of additives according to need. For example, the composition can be admixed with an antioxidant such as zinc thiophosphate, phenolic compounds, e.g. di-tert-butyl p-cresol, amine compounds, e.g. diphenyl amine, and the like. Other additives to be added to the inventive composition include detergent-dispersants of the types of sulfonates, phenates, phosphonates, imides and amides, molybdenum dithiophosphate, molybdenum dithiocarbamate, phosphorus-containing extreme-pressure additives, sulfur-based extreme-pressure additives, friction modifiers and other extreme-pressure additives and oiliness improvers, corrosion inhibitors, anti-foam agents, rust inhibitors and so on each in a limited amount.

It is further optional that the inventive lubricating oil composition is admixed with a viscosity-index improver and pour-point depressor such as polymethacrylates, copolymers of olefins, polybutenes and the like. It is noteworthy that the low-temperature viscosity of the composition can be improved

by the addition of a smaller amount of these additives than in the conventional lubricating oil compositions so that the decrease in the high-temperature cleanness by these additives can be minimized.

As is understood from the above given description, the lubricating oil composition of the present invention is imparted with greatly improved high-temperature cleanness and excellent friction characteristics with wet clutches. Further, the inventive lubricating oil composition well retains the excellent stability against oxidation and performance at extremely low temperatures as the inherently advantageous features of the poly- α -olefins along with still less expensiveness than the poly- α -olefins per se. Moreover, the inventive lubricating oil composition does not require addition of a large amount of a pour-point depressor or viscosity-index improver, which may have an adverse influence on the high-temperature cleanness of the lubricating oil, so that the high-temperature cleanness of the composition can be exhibited so much. Accordingly, the lubricating oil composition of the invention is very useful in a variety of applications where lubrication is required.

In the following, examples are given to illustrate the lubricating oil composition of the invention in more detail.

Preparation of Compositions 1 to 7.

Seven different base oils having a kinematic viscosity of 5 centistokes at 100°C were prepared each by uniformly blending the poly- α -olefin A₁ and one of the mineral oils B₁ to B₆

characterized by the property parameters indicated in Table 1 below in the weight proportion indicated in Table 2 below. The poly- α -olefin was a mixture of the dimer to tetramer of 1-decene.

Each of the base oils (kinematic viscosity of 5 centistokes at 100°C) was modified by the admixture of 7% by weight of a copolymer of ethylene and propylene having an average molecular weight of about 65,000 so as to be equivalent to an oil of the SAE viscosity grade 10W/30 and further admixed with 8% by weight of a commercial product of an additive package for engine oils containing calcium sulfonate as the principal ingredient. The thus prepared seven lubricating oil compositions are referred to as the Compositions 1 to 7 hereinbelow.

Preparation of Composition 8.

The base oil was prepared by uniformly blending the poly- α -olefin A₂, which was also a mixture of the dimer to tetramer of 1-decene, and the mineral oil B₅ characterized by the property parameters indicated in Table 1 in a 50:50 by weight proportion. The base oil was admixed with 15% by weight of the same ethylene-propylene copolymer as used in the formulation of the Compositions 1 to 7. The thus prepared lubricating oil composition is referred to as the Composition 8 hereinbelow.

Table 1

	Poly- α -olefin		Mineral Oil					
	A ₁	A ₂	B ₁	B ₂	B ₃	B ₄	B ₅	B ₆
Kinematic Viscosity (cSt) 100°C	5.0	4.0	5.0	11.2	5.0	5.0	4.0	10.9
Pour Point (°C)	-50>	-50>	-45	-42.5	-45	-20	-15	-15
%C _A	0.1>	0.1>	0.1>	0.2	6.5	5.5	7.3	7.0
Sulfur Content (ppm)	1>	1>	1>	1	25	10	500	6500

Table 2

		This Invention				Comparison			
		Composition				Composition			
		1	2	3	4	5	6	7	8
Poly- α -olefin	Kind	A ₁	A ₁	A ₁	A ₁	A ₁	A ₁	—	A ₂
	Amount (wt.%)	25	50	75	50	100	50	—	50
Mineral Oil	Kind	B ₁	B ₁	B ₁	B ₃	—	B ₄	B ₁	B ₅
	Amount (wt.%)	75	50	25	50	—	50	100	50

Examples 1 to 4 and Comparative Examples 1 to 4.

Each of the Compositions 1 to 8 was subjected to the test of the high-temperature cleanness according to the testing procedure described below to give the results shown in Table 3 below which also shows the kinematic viscosity at 100°C and the low-temperature viscosity at -30°C measured according to the procedure specified in ASTM D 2983.

[Test of high-temperature cleanness]

The sample oil was subjected to the panel-coking test according to the procedure specified in Federal Test Method

Standard No. 791a, Method 3462T with the conditions of the panel temperature of 320°C, oil temperature of 100°C and testing time of 3 hours and the high-temperature cleanness of the oil was evaluated by the weight increase of the panel in mg after the test.

Table 3

		High-temperature Cleanness (mg)	Low-temperature Viscosity (cp) (-30°C)	Kinematic Viscosity(cSt) (100°C)
Example 1	Composition 1	68	7,680	8.0
"	2	70	5,380	8.0
"	3	75	3,980	8.0
"	4	73	5,450	8.0
Comparative Example 1	"	125	3,000	8.0
"	2	78	27,500	7.9
"	3	86	11,200	8.1
"	4	155	26,500	9.5

Example 5 and Comparative Examples 5 and 6.

The Compositions 2, 6 and 8 prepared in the above described manner were each subjected to the test of stability against oxidation according to the procedure specified in JIS K 2514 to give the results of the increase in the overall acid value shown in Table 4 below in mg KOH/g together with the viscosity ratio at 100°C.

Table 4

		Viscosity Ratio (100°C)	Increase in Overall Acid Value (mg·KOH/g)
Example 5	Composition 2	1.25	0.2
Comparative Example 5	" 6	1.52	5.8
" 6	" 8	1.85	7.0

Preparation of Compositions 9 to 11.

The Compositions 9 to 11 were each prepared by admixing a base oil composed of the poly- α -olefin A₂ and one of the mineral oils characterized in Table 1 in the weight proportion indicated in Table 5 below with 10% by weight of a commercial product of an additive package for ATF containing an ashless dispersant as the principal ingredient and 5% by weight of a polymethacrylate having a weight-average molecular weight of about 80,000.

Table 5

		This Invention Composition 9	Comparison Composition 10	Composition 11
Poly- α - olefin	Kind	A ₂	A ₂	A ₂
	Amount (wt.%)	50	50	100
Mineral Oil	Kind	B ₁ /B ₂	B ₄ /B ₆	—
	Amount (wt.%)	40/10	40/10	—

Example 6 and Comparative Examples 7 and 8.

The Compositions 9 to 11 prepared in the above described manner were each subjected to the SAE No. 2 friction test under the conditions indicated below to give the results shown in Table 6 by the values of μ_0/μ_{1200} , in which μ_{1200} is the coefficient of dynamic friction at a velocity of rotation of 1200 rpm and μ_0 is the coefficient of static friction in a static condition.

[SAE No. 2 friction test]

The friction characteristics of the sample oil were evaluated using an SAE No. 2 testing machine manufactured by Greening Co., U.S.A., under the following experimental conditions.

Discs: 2 paper-based discs for automatic transmission

Plates: 3 steel-made plates for automatic transmission

Revolution of motor: 3000 rpm

Pressing-down pressure of piston: 10 kg/cm²

Temperature of oil: 100°C

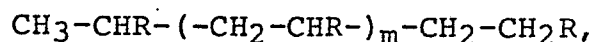
Table 6

				μ_0/μ_{1200}
Example 6	Composition 9			1.01
Comparative Example 7	"	10		1.05
"	8	"	11	1.10

WHAT IS CLAIMED IS:

1. A lubricating oil composition which comprises:
 - (A) from 15 to 85 parts by weight of a poly- α -olefin having a kinematic viscosity in the range from 1.5 to 150 centistokes at 100°C; and
 - (B) from 85 to 15 parts by weight of a mineral oil having a kinematic viscosity in the range from 2 to 50 centistokes at 100°C and a pour point of -35°C or below.

2. The lubricating oil composition as claimed in Claim 1 wherein the poly- α -olefin is a compound represented by the general formula:



in which R is an alkyl group having 4 to 12 carbon atoms and m is zero or a positive integer not exceeding 30.

3. The lubricating oil composition as claimed in Claim 1 wherein the poly- α -olefin is a dimer, trimer or tetramer of 1-decene or a mixture thereof.

4. The lubricating oil composition as claimed in Claim 1 wherein the mineral oil contains 20% by weight or less of aromatic hydrocarbon compounds and 50 ppm by weight or less of sulfur.