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(71) Applicant *(for all designated States except US)*: **SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.** [NL/NL]; Carel van Bylandtlaan 30, NL-2596 HR The Hague (NL).

(71) Applicant *(for US only)*: **SHELL OIL COMPANY** [US/US]; PO Box 2463, One Shell Plaza, Houston, Texas 77252-2463 (US).

(72) Inventors: **KAMATA, Kumiko**; 4052-2 Nakatsu, Aikawa-Cyo, Aikou-Gun, Aikou, Kanagawa 243-0303 (JP). **MORIKI, Keiichi**; 2-3-2, Diaba, Minato-ku, Tokyo, Tokyo 135-8074 (JP). **TAZAKI, Hiroyuki**; 4052-2 Nakatsu, Aikawa-Cyo, Aikou-Gun, Aikou, Kanagawa 243-0303 (JP).

(74) Agent: **MATTHEZING, Robert, Maarten**; Shell International B.V., P.O. Box 384, NL-2501 CJ The Hague (NL).

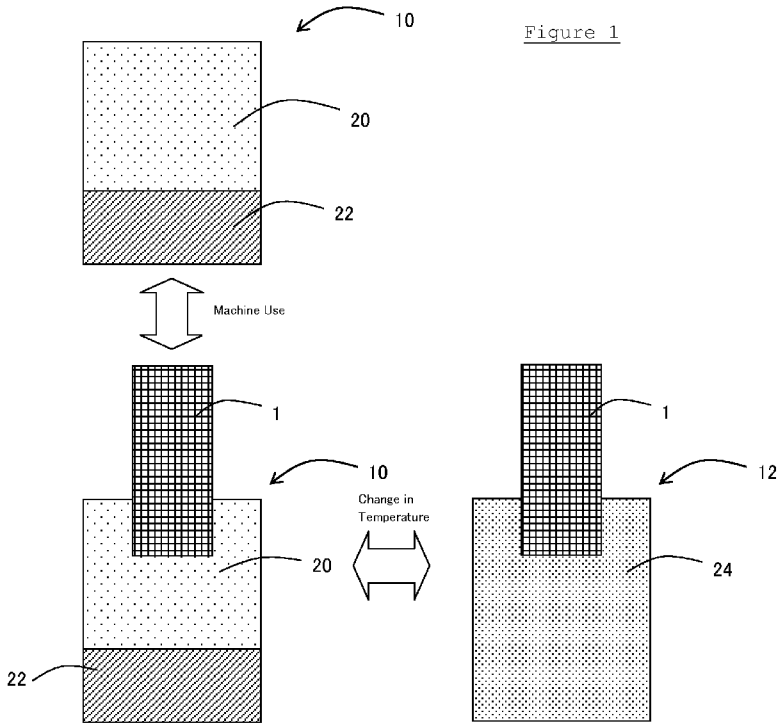
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(54) Title: A DRIVE SYSTEM TRANSMISSION LUBRICANT OIL COMPOSITION

Figure 1



(57) Abstract: A drive system transmission lubricant oil composition featuring superior fuel economy as well as wear resistance which features a suitable viscosity at high temperatures (kinematic viscosity at 100°C of 4.0- 7.0 mm^2/s) and manifests a reduction in viscosity at moderate to low temperatures and which furthermore achieves a low traction coefficient which is produced by mixing together (a) a low viscosity component (lubricant base oil) featuring a low density and low naphthene content, (b) a high viscosity component comprising polyalkylene glycol, and (c) a control component which features a specific ratio of oxygen to carbon by weight.

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A Drive System Transmission Lubricant Oil Composition

Field of the Invention

The present invention relates to a lubricant oil composition which contains, (a) a low viscosity component (lubricant base oil) featuring a low density and low naphthene content, (b) a high viscosity component comprising polyalkylene glycol, and (c) a control component which features a specific ratio of oxygen to carbon by weight, which furthermore is characterized in that it undergoes liquid phase separation of the low viscosity component and high viscosity component at an arbitrary temperature. The lubricant oil composition constituted by the present invention can be suitably used as a lubricant oil composition in transmission units used in automobiles, industrial applications, construction machinery or agricultural machinery.

Background of the Invention

In recent years, there has arisen a demand for the realization of increased fuel economy by increasing the power transmission rate - that is, decreasing power loss due to friction - as part of the functional properties of lubricant oil used in the lubrication of vehicle drive system transmission units.

In order to achieve these aforementioned increases in fuel economy, in the lubrication of a drive system transmission unit using the aforementioned lubricant oil, not only must an appropriate viscosity (oil film) be maintained at high temperature conditions (exceeding 60°C) to reduce pumping loss, under low to moderate temperature conditions (60°C or less) the viscosity of the lubricant oil must be reduced to improve agitation

resistance (Kurihara, Journal of the Japanese Society of Tribologists, 2009, 54, 4, 242 - 247). For lubricant oil used in current drive system transmission units, kinematic viscosity under high temperature conditions (100°C) must be set to approximately 5.0 mm^2/s (4.0 mm^2/s - 6.0 mm^2/s) or greater in order to maintain an oil film. Under these physical constraints, given performance trade-offs involving shear stability, etc. the sufficient addition of viscosity index improving polymer is not possible and either the viscosity index is left relatively low at approximately 170 or it becomes problematic to decrease viscosity in order to reduce agitation resistance arising due to viscosity at moderate to low temperatures.

To resolve the above issues, WO96/11244 discloses a lubricant oil which functions at both high and low temperatures, by combining a low viscosity lubricant oil as well as a high viscosity lubricant oil, which utilizes only the properties of the low viscosity lubricant oil at low temperatures while utilizing a property of the oil by which viscosity increases by mixing a high viscosity lubricant oil with a low viscosity lubricant oil at high temperatures. However, using this method, the kinematic viscosity and separation temperature are uniquely determined by the composition ratio and type of oils combined, so it is difficult to confer properties suited to applications in specific systems (for example, drive train transmission units).

Furthermore, reduction of the traction coefficient has been shown to be an effective means of achieving increased fuel economy when performing lubrication of drive system transmission units (in particular, drive system final reduction gear units) using the

aforementioned lubricant oils (Ueno et al, Preprinted Collection of the Annual Conference at the 2006 Fall Meeting of the Society of Automotive Engineers, No. 99 - 06, p. 3 - 6 (20065726)).

The present invention was developed in view of the aforementioned issues in order to provide a drive system transmission lubricant oil composition featuring a suitable viscosity at high temperatures (kinematic viscosity at 100°C of 3.5 - 7.0 mm^2/s) even when a viscosity index improving polymer is not added as well as reduced viscosity at moderate to low temperatures and furthermore achieves superior fuel economy (a low traction coefficient) as well as wear resistance.

Following extensive research to achieve the aforementioned objective, a drive system transmission lubricant oil composition featuring superior fuel economy as well as the aforementioned characteristics even when a viscosity index improving polymer is not added was successfully developed by mixing together (a) a low viscosity component (lubricant base oil) featuring a low density and low naphthene content, (b) a high viscosity component comprising polyalkylene glycol, and (c) a control component which features a specific ratio of oxygen to carbon by weight.

For the lubricant oil composition constituted by the present invention, because a combination of low viscosity and high viscosity components are used, liquid phase separation and precipitation (dispersion) of both the low viscosity and high viscosity components occurs at low temperatures, making the low viscosity component dominant and achieving a reduction in viscosity. Furthermore, at high temperatures, the low viscosity component and high viscosity component are combined, making it possible to

maintain a suitable viscosity.

Furthermore, by adding a specific amount of a compound featuring a specified ratio of oxygen to carbon by weight (for example, an aliphatic ester compound) as a control component, it is possible to freely adjust the temperature at which liquid phase separation of the aforementioned low viscosity component and high viscosity component occurs (the separation temperature). That is, by changing the type, physical properties, or amount added of the compound used as a control component, it is possible to adjust the separation temperature. Thus, it is possible to set the liquid phase separation temperature of the drive system transmission lubricant oil composition constituted by the present invention in accordance with applications or driving conditions corresponding to a variety of different operating temperatures.

Furthermore, the lubricant oil composition derived from the configuration of the present invention features good wear resistance, which is required of a drive system transmission lubricant oil composition.

Additionally, the drive system transmission lubricant oil composition constituted by the present invention achieves a reduction in the traction coefficient, which affects fuel consumption, by using a low viscosity component featuring specific properties.

Summary of the Invention

According to the present invention there is provided [1] - [9], below:

[1] A drive system transmission lubricant oil composition with a kinematic viscosity of 3.5 - 7.0 mm^2/s at 100°C produced by mixing the following: (A) A lubricant base oil having a viscosity of from 1.5 to 6.0 mm^2/s at 15°

and a density of from 0.750 to 0.830 g/cm^3 at 100°C containing at least one of the following: mineral oil, synthetic oil or GTL, as a low viscosity component, (B) from 3 to 35% by weight polyalkylene glycol (PAG) featuring an oxygen/carbon ratio of 0.450 to 0.580 by weight, as a high viscosity component, and (C) from 1 to 30% by weight of a compound featuring an oxygen/carbon ratio of from 0.080 to 0.350 by weight, as a control component.

[2] A drive system transmission lubricant oil composition as specified in [1] above in which the %Cp of the aforementioned low viscosity component ranges from 80 to 100.

[3] A drive system transmission lubricant oil composition as specified in [1] or [2] above which exists in a two-phase state when below its separation temperature and exists in a single-phase state when its separation temperature is exceeded, wherein the aforementioned separation temperature ranges from 60°C to 120°C.

[4] A drive system transmission lubricant oil composition as specified in any one of [1] - [3] above in which the aforementioned control component is an aliphatic ester featuring a carbon chain having from 4 to 18 carbon atoms, excluding the ester group.

[5] A drive system transmission lubricant oil composition as specified in any one of [1] - [4] above in which the density of the aforementioned high viscosity component at 20°C ranges from 1.000 to 1.050 g/cm^3.

[6] A drive system transmission lubricant oil composition as specified in any one of [1] - [5] above in which the density of the aforementioned control component at 20°C ranges from 0.800 to 1.000 g/cm^3.

[7] A drive system transmission lubricant oil composition

as specified in any one of [1] – [6] above in which the traction coefficient at 100°C is 0.0085 or less.

[8] A drive system transmission lubricant oil composition as specified in any one of [1] – [7] above which can be used in the lubrication of a drive transmission system for use in automobiles, industrial applications, construction machinery or agricultural machinery.

[9] A drive system transmission lubricant oil composition as specified in any one of [1] – [8] which can be used in a drive system final reduction gear unit.

Brief Description of Drawings

Figure 1: This figure shows a schematic diagram of the two-phase system constituted by the present invention (one example).

Figure 2: This figure shows one manifestation of separation temperature measurements for the lubricant oil constituting the present invention.

Detailed Description of the Invention

The lubricant oil composition constituted by the present invention is a lubricant oil composition featuring a suitable viscosity at high temperatures (100°C kinematic viscosity of 3.5 to 7.0 mm^2/s) even when a viscosity index improving polymer is not added as well as reduced viscosity at moderate to low temperatures and furthermore allows free control over separation temperature and achieves superior fuel economy (a low traction coefficient) as well as wear resistance, and thus is able to simultaneously provide superior fuel economy as well as wear resistance when used in the lubrication of a drive transmission system for use in automobiles, industrial applications, construction machinery or agricultural machinery.

Next, the present invention shall be described in

detail, but the present invention is in no way limited to the following specific applications and may be widely used in any number of applications.

The drive system transmission lubricant oil composition constituted by the present invention contains a low viscosity component, high viscosity component and control component which features properties intermediate to these two components. More specifically, the present invention pertains to a drive system transmission lubricant oil composition having a kinematic viscosity of from 3.5 to 7.0 mm^2/s at 100°C which includes the following:

(A) A lubricant base oil having a viscosity of from 1.5 to 6.0 mm^2/s at 15°C and a density of from 0.750 to 0.830 g/cm^3 at 100°C containing at least one of the following: mineral oil, synthetic oil or GTL, as a low viscosity component;

(B) from 3 to 35% by weight polyalkylene glycol (PAG) featuring an oxygen/carbon ratio of from 0.450 to 0.580 by weight, as a high viscosity component; and

(C) from 1 to 30% by weight of a compound featuring an oxygen/carbon ratio of from 0.080 to 0.350 by weight, as a control component.

The following section provides a description of the various components used as active ingredients followed by an explanation of the lubricant oil composition.

(A) Low Viscosity Component (Lubricant Base Oil)

A lubricant base oil of viscosity 1.5 to 6.0 mm^2/s at 15°C and density 0.750 to 0.830 g / cm3 at 100°C containing at least one of the following: mineral oil, synthetic oil or GTL, as a low viscosity component is used in the lubricant oil composition constituted by the present invention.

Here, the lubricant base oil pertaining to the present invention refers to anything which can be used as a base oil for a lubricant oil within the industry, which is a lubricant base oil containing at least one of the following: mineral oil, synthetic oil or GTL. For the present invention, examples of mineral oils, synthetic oils or GTLs include oils belonging to Groups 1 – 5 as defined under the base oil classification scheme of the API (American Petroleum Institute). Here, the base oil classification scheme of the API refers to broadly-defined categories for base oil materials established by the American Petroleum Institute in order to create guidelines for lubricating oil base oil.

For the present invention, the type of synthetic oil is not specified in any particular way, but examples of oils which are preferable include poly-α-olefins (PAOs) as well as hydrocarbon-based synthetic oils (oligomers). PAOs include both homopolymers and copolymers of an α-olefin. For example, the α-olefin may be a compound with a terminal double carbon-carbon bond, including butene, butadiene, hexene, cyclohexene, methyl cyclohexene, octene, nonene, decene, dodecene, tetradecene, hexadecene, octadecene, or eicosene. Valid examples of hydrocarbon-based synthetic oils (oligomers) include homopolymers or copolymers of ethylene, isobutene, or propylene. These compounds can be used either individually or as a mixture of two or more different compounds. Furthermore these compounds may feature any achievable isomeric structure, as long as a carbon-carbon double bond is present at the terminus, and both branching structures as well as straight chain structures are possible. Two or more types of the aforementioned structural isomers or double bond positional isomers can

also be used in concert. For these olefins, a carbon atom count of 5 or less results in a low flash point while a carbon atom count of 31 or greater results in high viscosity, limiting usefulness, so the use of a straight chained olefin containing from 6 to 30 carbon atoms is preferable.

For the present invention, commercially-available forms of PAO or hydrocarbon-based synthetic oil (oligomers) which can be obtained include Durasyn (Ineosu), Spectrasyn (ExxonMobil Chemical Company) and Lucant (Mitsui Petrochemical).

For the present invention, the type of mineral oil employed is not specified in any particular way, but preferable examples include paraffin or naphthene mineral oils obtained via the application of one or a combination of two or more of the following purification methods on the lubricating oil fraction obtained via vacuum distillation or atmospheric distillation of crude oil: solvent deasphalting, solvent extraction, hydrocracking, solvent dewaxing, catalytic dewaxing, hydrotreating, sulfuric acid washing, or clay treatment.

Furthermore, GTL (gas-to-liquid) synthesized via the Fischer-Tropsch process, which is a technique used to produce liquid fuel from natural gas, can be included as the low viscosity component constituted by the present invention. Compared to mineral oil base oils refined from crude oil, the sulfur and aromatic content of GTL is very low while the paraffin component ratio is very high, giving the latter superior oxidation stability and significantly reducing evaporative loss, so it can be preferably used as a base oil constituting the present invention.

Kinematic Viscosity and Density of the Low Viscosity

Component

Because the kinematic viscosity at 100°C of the lubricant base oil which serves as the low viscosity component pertaining to the present invention is responsible for maintaining a suitable oil film while also improving fuel economy and wear resistance, kinematic viscosity should range from 1.5 to 6.0 mm^2/s, preferably, from 3.0 to 6.0 mm^2/s, more preferably from3.5 to 6.0 mm^2/s, most preferably from 4.0 to 5.5 mm^2/s. Furthermore, the density at 15°C of the lubricant base oil which serves as the low viscosity component pertaining to the present invention should preferably range from 0.750 to 0.830 g/cm^3, more preferably from 0.790 to 0.830 g/cm^3, even more preferably from 0.800 to 0.829 g/cm^3. Additionally, the %Cp of the lubricant base oil which serves as the low viscosity component pertaining to the present invention should preferably range from 80 to 100, or, more preferably from 82 to 100, or, even more preferably, from 84 to 100. Thus, by utilizing a low density and high %Cp low viscosity component, it is possible to obtain a lubricant oil composition featuring superior fuel economy and wear resistance in which the apparent viscosity index (VI) is increased and the traction coefficient is decreased.

(B) High Viscosity Component (Polyalkylene Glycol (PAG) Featuring an Oxygen/Carbon Ratio of from 0.450 to 0.580 by Weight)

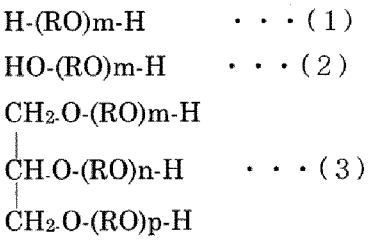
For the present invention, polyalkylene glycol (PAG) featuring an oxygen/carbon ratio of from 0.450 to 0.580, preferably from 0.450 to 0.500, more preferably from 0.450 to 0.470, which does not substantially mix with the low viscosity component at low temperatures but does mix at high temperatures can be used as the high viscosity

component used in conjunction with the aforementioned lubricant base oil which serves as the low viscosity component.

Ratio of Oxygen/Carbon by Weight

Here, the ratio of oxygen/carbon by weight represents the ratio of the weight of oxygen to the weight of carbon present in the component in question and this value primarily affects the physical properties, such as the density as well as the polarity, of the compound. For example, though polarity is also affected by the presence of functional groups such as ether groups, ester groups, hydroxyl groups, and carboxyl groups, because oxygen atoms are highly electronegative, there is a generally a tendency for polarity to increase as the ratio of oxygen/carbon by weight increases. With regard to density, because oxygen is heavier than carbon, compounds with a larger ratio of oxygen to carbon by weight generally tend to feature a higher density. Measurement of the ratio of oxygen to carbon by weight can be carried out according to the JPI-5S-65 (Experimental Methods for the Carbon, Hydrogen and Nitrogen Components of Petroleum Products) or JPI-5S-68 (Experimental Methods for the Oxygen Component of Petroleum Products) standards.

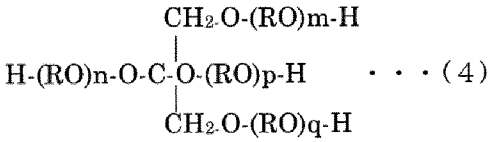
Examples of polyalkylene glycol (PAG), featuring an oxygen/carbon ratio of 0.450 to 0.580 by weight, which can be used in the lubricant oil composition constituted by the present invention include the compounds shown in Generalized Formulae (1) - (4) below;



$$H\text{-}(RO)m\text{-}H \quad \cdots(1)$$

$$HO\text{-}(RO)m\text{-}H \quad \cdots(2)$$

$$\begin{array}{l} CH_2\text{-}O\text{-}(RO)m\text{-}H \\ | \\ CH\text{-}O\text{-}(RO)n\text{-}H \\ | \\ CH_2\text{-}O\text{-}(RO)p\text{-}H \end{array} \quad \cdots(3)$$



$$H\text{-}(RO)n\text{-}O\text{-}C(CH_2\text{-}O\text{-}(RO)m\text{-}H)(CH_2\text{-}O\text{-}(RO)q\text{-}H)\text{-}O\text{-}(RO)p\text{-}H \quad \cdots(4)$$

Here, each instance of *R* indicates a straight chain or branched chain hydrocarbon group which is from 2 to 10 carbons in length, preferably from 2 to 8 carbons in length, more preferably from 2 to 6 carbons in length and *m* is an integer ranging from 2 to 500, preferably from 2 to 400 more preferably from 2 to 300. Note that there is no requirement that all instances of *R* are the same alkylene and a combination of different types of alkylenes is permitted. For example, in the case of a block copolymer of alkylene oxide featuring two types of (R1O)m as shown above, for (R1O)m above, both (R1-1O)m-1 and (R1-2O)m-2 can be specified.

Examples of polyalkylene glycols (PAGs) featuring an oxygen/carbon ratio of 0.450 to 0.580 by weight include compounds obtained via the addition polymerization of alkylene oxide to alcohol. For the alkylene oxide used as a raw material, only one type or two or more types may be used. Here, examples of monomer components which can be added include compounds which employ ethylene oxide, propylene oxide or butylene oxide, either individually or as a combination of two or more (for example, ethylene oxide / propylene oxide).

Kinematic Viscosity and Density of the High Viscosity Component

The kinematic viscosity at 100°C of the polyalkylene glycol (PAG) featuring an oxygen/carbon ratio of from 0.450 to 0.580 by weight which serves as the high viscosity component pertaining to the present invention should range from 2.5 to 100 mm^2/s, preferably from 2.5 to 80 mm^2/s more preferably from 2.5 to 70 mm^2/s. Furthermore, the density at 20°C of the aforementioned polyalkylene glycol (PAG) pertaining to the present invention should range from 1.000 to 1.050 g/cm^3, preferably from 1.000 to 1.020 g/cm^3, more preferably from 1.000 to 1.010 g/cm^3. By using a high viscosity component featuring this kinematic viscosity and density, it is possible to set the separation temperature of the liquid-liquid separation oil constituted by the present invention to a suitable temperature range. Note that it is also possible to combine and use two or more different high viscosity components.

(C) Control Component (Compound Featuring an Oxygen/Carbon Ratio of from 0.080 to 0.350 by weight)

In the lubricant oil composition of the present invention, a compound featuring an oxygen/carbon weight ratio of from 0.080 to 0.350, preferably, from 0.080 to 0.300, more preferably from 0.080 -to 0.250 can be used as a control component. The control component refers to a component which ensures that, in its presence, the low viscosity component and high viscosity component do not substantially mix at low temperatures but mix homogeneously at high temperatures. Note that it is also possible to combine and use two or more different control components. Here, the control component is not limited in any particular way as long as it is a compound featuring an oxygen/carbon weight ratio in the aforementioned range, but from the standpoint of polarity

and viscosity, among such compounds, compounds containing ester groups (ester compounds), for example, should preferably be used. Compounds which can be preferably used as compounds containing an ester group include aliphatic ester compounds containing a straight chain or branched chain hydrocarbon component as well as an ester functional group or aromatic ester compounds containing an aromatic component as well as an ester functional group. Even more preferable are aliphatic ester compounds (for example, aliphatic ester compounds with a carbon chain length of from 4 to 18, preferably from 4 to 16 even more preferably from 4 to 14 carbon atoms in length, excluding the ester group) and/or aromatic ester compounds which contain only carbon, hydrogen and oxygen as constituent elements.

Monoesters, diesters and triesters can be preferably used as the aforementioned ester compounds, and diesters are even more preferred. Examples of monoesters include esters of monovalent carboxylic acid (for example, formic acid, acetic acid, propionic acid, butyric acid, valeric acid, caproic acid, enanthic acid, caprylic acid, pelargonic acid, capric acid, undecyl acid, lauric acid, tridecyl acid, hexadecyl acid, heptadecyl acid and stearic acid) and monovalent alcohol (for example, straight or branched monovalent alcohols such as methanol, ethanol, propanol, butanol, pentanol, hexanol, heptanol, octanol, nonanol, and decanol). Examples of diesters include esters of dicarboxylic acid (for example, straight chain or branched chain carboxyllic acids such as malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid) and monovalent alcohol (for example, the aforementioned monovalent alcohols) or esters of

monovalent carboxyllic acid (for example, the aforementioned carboxyllic acids) and divalent alcohol (for example, straight chain or branched chain divalent alcohols such as ethylene glycol, propylene glycol, butylene glycol, pentylene glycol or hexylene glycol). Examples of triesters include esters of monovalent carboxylic acid (for example, the aforementioned monovalent carboxyllic acids) and trivalent alcohol (for example, glycerin and butane triol) or esters of trivalent carboxyllic acid (for example, citric acid and isocitric acid) and monovalent alcohol (for example, the aforementioned monovalent alcohols). More specifically, fatty acid diesters [for example, diisononyl adipic acid (trade name: DINA; Taoka Chemical)], fatty acid monoesters [for example, isooctyl stearate (trade name: EXCEPARL EH-S; Kao)], trimellitic acid esters [for example, trimellitic acid tri-normal-alkyl (trade name: TRIMEX N-08; Kao)] or fatty acid triesters [for example, oleic acid trimethylol propyl (trade name: Kaorubu 190; Kao)] can be preferably used.

Kinematic Viscosity and Density of the Control Component

For the compound featuring an oxygen/carbon weight ratio of from 0.080 to 0.350 which serves as the control component pertaining to the present invention, kinematic viscosity at 40°C should range from 5 to 75 mm^2/s, preferably from 7 to 60 mm^2/s, more preferably from 9 to 50 mm^2/s, kinematic viscosity at 100°C should range from 2.5 to 18 mm^2/s, preferably from 2.7 to 15 mm^2/s, more preferably from 2.8 to 10 mm^2/s, and density at 20°C should range from 0.800 to 1.010 g/cm^3, preferably from 0.830 to 1.005 g/cm^3, more preferably from 0.850 to 1.000 g/cm^3. By using a control component and high viscosity component featuring these densities, it is possible to

set the separation temperature of the liquid-liquid separation oil to a suitable temperature range.

Optional Components

One or more of the following optional additives can be used in the drive system transmission lubricant oil composition of the present invention as needed: anti-wear agents, rust inhibitors, metal inertness agents, hydrolysis inhibitors, antistatic agents, defoamers, antioxidants, dispersants, detergents, extreme pressure agents, friction modifiers, viscosity index improvers, pour point depressants, thickeners, metal detergents, ashless dispersants or corrosion inhibitors. For example, the use of an "additive package" which is used to increase performance (for example, various packages such as a sulfur-phosphorous package or ATF additive package) is possible.

The Overall Composition of the Drive System Transmission Lubricant Oil Composition

The drive system transmission lubricant oil composition of the present invention contains:
3 to 35% by mass, or, preferably, 5 to 30% by mass, or, even more preferably, 10 to 25% by mass polyalkylene glycol (PAG) featuring an oxygen/carbon weight ratio of 0.450 to 0.580 which serves as a high viscosity component (B); and

From 1 to 30% by mass,, preferably from 2 to 25% by mass, even more preferably from 3 to 25% by mass of a compound featuring an oxygen/carbon weight ratio of from 0.080 to 0.350 which serves as a control component (C), relative to the entire mass (100% by mass) of the lubricant oil composition. Furthermore, the composition may contain, for example, from 1 to 25% by mass optional components relative to the total mass of the lubricant

oil composition.

Additionally, the composition can be made to contain a lubricant base oil which serves as the low viscosity component (A) to bring the total mass up to 100% by mass. The amount of low viscosity component (A) included in the overall composition is not limited in any particular way but the amount of low viscosity component (A) should preferably be from 34 to 95% by mass, more preferably from 44 to 94% by mass, even more preferably from 49 to 86% by mass relative to the entire mass of the lubricant oil composition (100% by mass).

Viscosity of the Drive System Transmission Lubricant Oil Composition

By adding a control component featuring an oxygen/carbon weight ratio of from 0.080 - 0.350 to the drive system transmission lubricant oil composition constituted by the present invention, the low viscosity component and high viscosity component are separated into two phases at low temperature, but as temperature is increased, the low viscosity and high viscosity components mix and become a single phase above the separation temperature. Since typically the machinery to be lubricated comes into close contact with the liquid surface of the lubricant oil, the viscosity of the low viscosity component which is typically part of the upper phase should preferably make a contribution at lower temperatures and the kinematic viscosity at 40°C should preferably be from 5 to 100 mm^2/s, more preferably from 8 to 50 mm^2/s, even more preferably from 10 to 40 mm^2/s. Here, kinematic viscosity at 40°C is measured by heating up the lubricant oil composition until it is uniform after which the composition is cooled until it separates into two phases and the upper phase is measured.

Therefore, as a result of heating and then cooling the composition, part of the control component is mixed into the low viscosity component phase. Conversely, at higher temperatures, the viscosity of a homogeneous mixture of the low viscosity component and high viscosity component makes a contribution and the kinematic viscosity at 100°C ranges from 3.5 to 7.0 mm^2/s, preferably from 4.0 to 7.0 mm^2/s, more preferably from 4.0 to 6.5 mm^2/s, even more preferably from 4.0 to 6.0 mm^2/s.

The apparent viscosity index (VI) of the lubricant oil composition constituted by the present invention should preferably range from 100 to 300, while from 150 to 250 is more preferable and from 180 to 230 is even more preferable. The viscosity index is a convenient index which indicates the degree of change in viscosity of the lubricating oil caused by a given change in temperature. The viscosity index as it pertains to the present invention can be computed according to the viscosity index computation method specified in the JISL2283 standard using the viscosity at 40°C of a sample oil (upper phase produced following separation into two phases) as well as the viscosity at 100°C of another sample oil (the lubricant oil composition after merging to produce a single phase). A high viscosity index indicates that changes to viscosity in response to changes in temperature are small.

For the present invention, by adding a control component featuring an oxygen/carbon weight ratio of from 0.080 - 0.350, it is possible to arbitrarily reduce the separation temperature of the composition. Therefore, the present invention also provides a method for controlling the separation temperature of the lubricant oil composition.

Separation Temperature

As described above, the lubricant oil composition constituted by the present invention features a separation temperature at which it transitions from a single-phase state to a two-phase state. Here, the separation temperature refers to the temperature at which cloudiness (precipitate) becomes visible during cooling following heating of the lubricant oil composition when it is in a two-phase state to convert it to a single-phase state. The lubricant oil composition constituted by the present invention should preferably be mixed such that the high viscosity component increases the viscosity of the low viscosity component at high temperatures (the low viscosity and high viscosity components should preferably be homogeneous). A suitable lubricant oil composition constituted by the present invention should separate into two phases at 60°C or below and converge to a single phase (homogeneity) when the separation temperature (60°C to 120°C) is exceeded. Furthermore, the lubricant oil composition constituted by the present invention can be arbitrarily controlled to a desired separation temperature.

Contribution of the Control Component

The control component functions to set the desired value of the separation temperature at which a transition from a single phase to two phases occurs, which should preferably range from 60°C to 120°C, more preferably from 65°C to 110°C, even more preferably from 70°C to 100°C; and preferably in a lubricant oil which separates into two phases at 60°C or below and converges to a single phase (homogeneity) when the separation temperature (60°C to 120°C) is exceeded. Furthermore, at low temperatures, the control component can exist as a separate phase even

when part of, or the entirety of, the control component is mixed into the upper phase and/or the lower phase. Therefore, when the control component is mixed into the upper phase and/or the lower phase while at low temperature, the control component functions as a component which is capable of changing the original viscosities of the low viscosity component which constitutes the primary component of the upper phase and/or the high viscosity component which constitutes the primary component of the lower phase. Therefore, if the control component is mixed into the upper phase and/or the lower phase while at a low temperature and if the viscosity of the low viscosity component is lower than that of the control component which is in turn lower than that of the high viscosity component, the viscosity of the low viscosity component which constitutes the primary component of the upper phase is less than the viscosity of the upper phase and the viscosity of the lower phase is lower than the viscosity of the high viscosity component which constitutes the primary component of the lower phase.

Traction Coefficient

The lubricant oil composition constituted by the present invention should preferably feature a traction coefficient at 100°C which is 0.0085 or less. By featuring such a traction coefficient, the composition can be used in the lubrication of a drive system transmission unit for use in automobiles, industrial applications, construction machinery or agricultural machinery, and, more specifically, can be used as a lubricant oil for transmission units, particularly a drive system final reduction gear unit. The traction coefficient at 100°C should preferably be 0.0080 or less,

or, even more preferably, 0.0076 or less. Furthermore, a lower limit for the traction coefficient is not specified in any particular way, but for practical purposes a traction coefficient at 100°C of 0.0040 or greater, for example can be used as an acceptable range.

Wear Resistance

The lubricant oil composition constituted by the present invention should preferably yield a wear track diameter of 0.50 mm or less in a shell 4-ball wear test performed at an oil temperature of 90°C, with a load of 40 kgf and rotational velocity of 1800 rpm over 60 minutes according to the ASTM D4172 standard. By featuring such wear resistance, the composition can be used in the lubrication of a drive system transmission unit for use in automobiles, industrial applications, construction machinery or agricultural machinery, and, more specifically, can be used as a lubricant oil for transmission units, particularly a drive system final reduction gear unit. The wear track diameter produced via the aforementioned test should preferably be 0.45 mm or less, and a diameter of 0.39 mm or less is even more preferable. Furthermore, a lower limit for the wear track diameter produced via the aforementioned test is not specified in any particular way, but for practical purposes a wear track diameter produced by the aforementioned test of 0.30 mm or greater, for example, can be used as an acceptable range.

Examples Illustrating Aspects of Actual Use of the Lubricant Oil

First, an example showing aspects of the invention when starting use of a machine will be explained with reference to Figure 1. Figure 1 (top) represents one aspect of the lubricant oil composition constituted by

the present invention and shows Two-Phase State 10 which is the composition's low-temperature state. Because Low Viscosity Component 20 is a low-viscosity lubricant oil, it resides in the upper phase and because High Viscosity Component 22 is a high-viscosity lubricant oil, it resides in the lower phase. Figure 1 (lower left) shows a state in which Machine 1, which is being lubricated, is used and the machine is immersed in the upper phase of the lubricant oil composition. During startup (low temperature), Low Viscosity Upper Phase 20 is the principal contributor to lubrication, while High Viscosity Lower Phase 22 barely contributes to lubrication. Because the low viscosity lubricant oil provides sufficient lubrication performance (viscosity) at low temperature, lubrication performance is not impeded even when only low viscosity components are present. Figure 1 (lower right) shows Single-Phase State 12 which is produced following an increase in temperature resulting from continued use of the machine.

Here, as a result of an increase in temperature, Low Viscosity Component 20 and High Viscosity Component 22 mix, producing Homogeneous Lubricant Oil Composition 24. A decrease in viscosity which accompanies an increase in the temperature of Low Viscosity Component 20 is ameliorated by high viscosity component 22 to a greater extent than when only Low Viscosity Component 20 is used, due to mixing in of High Viscosity Component 22, so even when an increase in temperature occurs, issues such as collapse of the oil film do not occur. Because the composition becomes a homogeneous single-phase system when the separation temperature is exceeded, a decrease in the viscosity of the low viscosity component is compensated for by the high viscosity component.

One of the unique features of the present invention is the behavior of the lubricant oil composition produced by a mixture of a low viscosity component and high viscosity component. In particular, at low temperatures, a low viscosity lubricant oil, such as a hydrocarbon, which is typically present in the upper phase, contributes to the lubrication of the machine while at high temperatures a mixture of high viscosity lubricant oil and low viscosity lubricant oil contributes. When this is the case, by using a control component as specified in the present invention, it is possible to maintain a nearly comparable kinematic viscosity at high temperature even when the separation temperature is reduced. On the other hand, as shown in WO96/11244, if the ratio of low viscosity to high viscosity components is simply changed, a correlation between kinematic viscosity and separation temperature is not necessarily observed, so the design of a composition with a kinematic viscosity and separation temperature suited to a specific application or environment is extremely difficult.

Applications

The lubricant oil composition constituted by the present invention can be used as a lubricant oil composition for a drive system transmission unit. For example, the composition can be applied to the lubrication of drive system transmission units for use in automobiles, industrial applications, construction machinery or agricultural machinery. More specifically, the composition can be used as a lubricant oil for transmission units (gear unit, CVTs, ATs, MTs, DCTs, DT's, etc.) and in particular drive system final reduction gear units.

Examples

Next the present invention shall be further described by way of examples, but the present invention is not limited to these examples.

Test Methods

Data Measurements

Data measurements for the lubricant oil composition constituted by the present invention as well as lubricant oil compositions used in comparative examples were performed according to the following method.

[1] Separation Temperature

Separation temperature was measured using a CORNING PC-420D as a heater (see Figure 2).

(1) 50 g of sample was placed in a 100 ml beaker and Stirring Bar 100 was placed inside the sample.

(2) As shown in Figure 2, an experimental apparatus was established by placing Thermocouple 102 (to measure oil temperature), to which Thermometer 101 was connected, inside the oil.

(3) The stirring speed of Hot Stirrer 103 was then set to 300 rpm.

(4) Plate temperature was then set to 200°C and the oil was heated until the oil temperature reached 120°C.

(5) Once the oil temperature reached 120°C, heating was stopped and the sample was cooled to close to room temperature.

(6) The oil temperature was then increased to 120°C via heating, as described in Step (4).

(7) Once oil temperature reached 120°C, heating was stopped and the state of the sample inside the beaker was observed.

(8) When the sample inside the beaker became cloudy (when precipitate became visible), the oil temperature was recorded as the separation temperature. Although the

measurement method was visual in nature, reference was made to aniline point measurement (JIS K 2256).

[2] Dynamic Viscosity (40°C)

Measurements of kinematic viscosity (40°C) were performed according to the method outlined in JIS K 2283 using an Ubbelohde viscometer as a testing apparatus. Because the lubricant oil composition produced by mixing the three components together was capable of separating into two phases at the measurement temperature, the supernatant portion of the upper phase (wherein the low viscosity component constituted the primary component) was taken and used as a sample for measuring viscosity.

[3] Dynamic Viscosity (100°C)

Measurements of kinematic viscosity (100) were performed according to the method outlined in JIS K 2283 using an Ubbelohde viscometer as a testing apparatus. The sample was collected into a viscosity tube after being heated in advance to 100°C and placed in a bath before the temperature decreased after which measurements were performed.

[4] Viscosity Index (VI) and Apparent Viscosity Index (Apparent VI)

Viscosity index (VI) and apparent viscosity index (apparent VI) were computed from 40°C and 100°C kinematic viscosity data as shown above according to the method outlined in JIS K 2283. Note that, the apparent VI of the lubricant oil composition obtained by mixing the three components, unlike the standard VI, is obtained measuring the kinematic viscosity at 40°C using a supernatant constituting part of the composition.

[5] Density (15°C)

Measurements of density (15°C) were performed according to the method outlined in JIS K 2249 using a

vibration type testing apparatus (Kyoto Electronics Manufacturing: DA-300).

[6] Ratio of Oxygen to Carbon by Weight

Measurement of the ratio of oxygen to carbon by weight (the ratio of the weight of oxygen to the weight of carbon) was carried out according to the JPI-5S-65 (Experimental Methods for the Carbon, Hydrogen and Nitrogen Components of Petroleum Products) or JPI-5S-68 (Experimental Methods for the Oxygen Component of Petroleum Products) standards using an Elementar Vario EL III as a testing apparatus.

[7] %Ca, %Cn and %Cp

%Ca, %Cn and %Cp values were obtained according to the method outlined in ASTM D3238.

[8] Traction Coefficient at 100°C

For the traction coefficient, an inter-ball traction coefficient was measured using an EHL Rolling and Sliding Friction Measuring Device (PCS Instruments; EHL Ultra Thin Film Measurement System) using an SUJ disk and diameter (φ) of 19.05 mm. Measurements were carried out under the following conditions: oil temperature: 100°C; load: 40N; circumferential velocity: 3 m/s; and slip rate: 30%.

[9] Shell 4-Ball Wear Test

A shell 4-ball wear test was performed at an oil temperature of 90°C, load of 40 kgf and rotational velocity of 1800 rpm over 60 minutes according to the ASTM D4172 standard.

Examples and Comparative Examples

In the following Examples and Comparative Examples, a lubricant composition was prepared using the following components. Unless otherwise specified, amounts are expressed as amounts per unit weight. The components

used in the Examples and Comparative Examples were as follows.

[1] Low Viscosity Component (Lubricant Base Oil)

Low Viscosity Components 1 – 5, shown below, were used as low viscosity components. The composition of the low viscosity components used is shown in Table 1. Note that the oxygen / carbon ratio by weight for these components was in all cases 0 (as no oxygen atoms were present). Table 1 shows the properties of the low viscosity components measured according to the aforementioned methods – in particular, the kinematic viscosity at 100°C, viscosity index (VI), density (15°C) and %Cp value.

(1) "Low Viscosity Component 1" was produced by mixing GTL Base Oil 1 (Royal Dutch Shell Co.), classified as API base oil category: Group 3 and featuring a kinematic viscosity at 40°C of 18.2 mm^2/s, kinematic viscosity at 100°C of 4.1 mm^2/s, and density at 15°C of 0.817 g/cm^3, with GTL Base Oil 2 (Royal Dutch Shell Co.), classified as API base oil category: Group 3 and featuring a kinematic viscosity at 40°C of 44.2 mm^2/s, kinematic viscosity at 100°C of 7.6 mm^2/s, and density at 15°C of 0.828 g/cm^3, in the composition ratio (by percent mass) specified in Table 1.

(2) "Low Viscosity Component 2" was produced by mixing Synthetic Oil 1 (Exxon Mobil Chemical Co., Ltd.; marketed as Spectra Syn 4; generic name: PAO4), classified as API base oil category: Group 4 and featuring a kinematic viscosity at 40°C of 17.6 mm^2/s, kinematic viscosity at 100°C of 4.0 mm^2/s, and density at 15°C of 0.820 g/cm^3, with Synthetic Oil 2 (BP; marketed as PAO8), classified as API base oil category: Group 4 and featuring a kinematic viscosity at 40°C of 46.6 mm^2/s, kinematic

viscosity at 100°C of 7.8 mm^2/s, and density at 15°C of 0.831 g/cm^3, in the composition ratio (by percent mass) specified in Table 1.

(3) "Low Viscosity Component 3" was produced by mixing Synthetic Oil 1 and Synthetic Oil 2, as described above, as well as Mineral Oil 1 (SK Lubricants; marketed as Yubase-4), classified as API base oil category: Group 3 and featuring a kinematic viscosity at 40°C of 19.4 mm^2/s, kinematic viscosity at 100°C of 4.3 mm^2/s, and density at 15°C of 0.833 g/cm^3, with Mineral Oil 2 (SK Lubricants; marketed as Yubase-8), classified as API base oil category: Group 3 and featuring a kinematic viscosity at 40°C of 45.8 mm^2/s, kinematic viscosity at 100°C of 7.5 mm^2/s, and density at 15°C of 0.845 g/cm^3, in the composition ratio (by percent mass) specified in Table 1.

(4) "Low Viscosity Component 4" was produced by mixing Mineral Oil 1 and Mineral Oil 2, as described above, in the composition ratio (by percent mass) specified in Table 1.

(5) "Low Viscosity Component 5" was composed of only Mineral Oil 3 (Showa Shell Sekiyu Co., Ltd.), classified as API base oil category: Group 1 and featuring a kinematic viscosity at 40°C of 24.4 mm^2/s, kinematic viscosity at 100°C of 4.6 mm^2/s, and density at 15°C of 0.865 g/cm^3 (100 % by mass).

[2] Additives

A "sulfur-phosphorus additive package" was included in the control component as an additive. The sulfur-phosphorus additive package was formulated with sulfurized olefins and phosphate amine salts, in addition to other components, with a phosphorus content of approximately 1.2% by mass and sulfur content of

approximately 36% by mass. The additive package included a combination of additives used to improve performance, including friction modifiers, antioxidants, rust inhibitors, antiwear agents, dispersing agents and detergents.

[3] Control Component

A fatty acid diester (Taoka Chemical Co.; adipic acid diisononyl, marketed as DINA), featuring a density at 20°C of 0.924 g/cm^3, oxygen/carbon ratio by weight of 0.221, kinematic viscosity at 40°C of 10.81 mm^2/s and kinematic viscosity at 100°C of 3.042 mm^2/s was used as a control component.

[4] High Viscosity Component

Polyalkylene glycol and propylene oxide (NOF Co.; marketed as Uni-Lube MB-700), featuring a density at 20°C of 1.003 g/cm^3, oxygen / carbon ratio by weight of 0.451, kinematic viscosity at 40°C of 616 mm^2/s and kinematic viscosity at 100°C of 92.73 mm^2/s was used as a high viscosity component.

Examples 1 - 3 and Comparative Examples 1-2

The high viscosity component, additives, control component and low viscosity component were weighed out, in that order, into a beaker based on the composition ratio (percent by mass) shown in Table 2 and stirring was performed to yield each sample lubricant oil composition (Examples 1-3 and Comparative Examples 1-2). The formulation of these lubricant oil compositions was determined in such a way that they would constitute a homogeneous single phase at 100°C, that is, such that the separation temperature was less than 100°C.

Measurements of kinematic viscosity at 40°C (mm^2/s), kinematic viscosity at 100°C (mm^2/s), (apparent) viscosity index, separation temperature (°C) and traction

coefficient at 100°C were carried out for each lubricant oil composition thus prepared and the results are shown in Table 2.

Table 1

Table 1: Composition of the Low Viscosity Components Used						
	Name of Base Oil Prior to Mixing	Low Viscosity Component 1	Low Viscosity Component 2	Low Viscosity Component 3	Low Viscosity Component 4	Low Viscosity Component 5
Low Viscosity Component Composition (Content: Percent by Mass)	GTL Base Oil 1	80				
	GTL Base Oil 2	20				
	Synthetic Oil 1 (PAO4)		80	40		
	Synthetic Oil 2 (PAO8)		20	10		
	Mineral Oil 1 (Yubase-4)			42.5	85	
	Mineral Oil 2 (Yubase-8)			7.5	15	
	Mineral Oil 3					100
Low Viscosity Component	Kinematic Viscosity	4.6	4.6	4.6	4.6	4.6

Properties	at 100 °C (mm^2/s)					
	VI	135	130	130	130	107
	Density @ 15 °C (g/cm^3)	0.8193	0.8225	0.8287	0.8348	0.8626
	%Ca	0	0	0	0	0
	%Cn	8.3	9.4	15.6	21.8	28.8
	%Cp	91.7	90.6	84.4	78.2	71.2

Table 2

Table 2: Traction Coefficient and Wear Resistance Experimental Results								
				Examples			Comparative Examples	
				1	2	3	1	2
	Lubricant Oil Composition Formulation	Low Viscosity Component	Low Viscosity Component 1 (Density at 15°C: 0.8193 g/cm^3)	80.12				
			Low Viscosity Component 2 (Density at 15°C: 0.8225 g/cm^3)		80.12			
			Low Viscosity Component 3 (Density at 15°C: 0.8287 g/cm^3)			80.12		
			Low Viscosity Component 4				80.12	

		(Density at 15°C: 0.8348 g/cm^3)							80.12
		Low Viscosity Component 5 (Density at 15°C: 0.8626 g/cm^3)							
			Control Component	10.88	10.88	10.88	10.88		10.88
			High Viscosity Component	6	6	6	6		6
			Additive Package	3	3	3	3		3
	Lubricant Oil Composition Properties	Kinematic Viscosity @ 40°C (mm^2/s)		20.4	20.6	20.2	20.7		23.1
		Kinematic Viscosity @ 100°C (mm^2/s)		5.5	5.4	5.4	5.4		5.5
		Viscosity Index		231	218	223	219		192
		Separation Temperature (°C)		84.0	83.3	77.4	80.2		47.1
		Traction Coefficient (Oil Temp: 100°C, Load: 40N, Sliding Speed: 3 m/s, Slip Ratio: 30%)		0.0069	0.0076	0.0076	0.0094		0.0115
		Shell 4-Ball Wear Test / Wear Track Diameter (mm) (Oil Temp: 90°C, Load: 40 kgf, Rotational Velocity: 1800 rpm, Measurement Duration: 60 min)		0.39	0.38	0.37	0.40		0.39

Based on these results, it was demonstrated in Examples 1-3 as well as Comparative Examples 1 and 2 that the composition featured a high (apparent) VI and sufficient wear resistance. For lubricant oils currently used in drive system transmission units, it is not possible to add a sufficient amount of viscosity index improving polymer due to performance trade-offs involving shear stability, etc., and when a viscosity index improving agent is not added, the viscosity index remains approximately 170. The lubricant oil compositions constituted from the Examples presented in the present application show viscosity indices of 200 or greater without the addition of a polymer. Furthermore, when a polymer is used to formulate a lubricant oil with both low viscosity and high viscosity indices without regard for shear stability, it is necessary to use an ultra-low viscosity base oil which is not employed in conventional lubricant oils, so there is an issue in which the composition becomes a non-usable lubricant oil featuring a low flash point and high volatility. However, the lubricant oil compositions constituted from the Examples of the present invention can be configured with base oils in common use, so it is possible to design a highly usable lubricant oil composition which features low volatility and a high flash point. Moreover, it was determined that the lubricant oil composition constituted by the present invention featured sufficient wear resistance when used in a drive system transmission unit or the like at a temperature at which the composition transitions to a single-phase state.

Furthermore, since the lubricant oil composition constituted by the present invention contains a control component in addition to a low viscosity component and

high viscosity component, it is possible to freely adjust the separation temperature of the lubricant oil composition between 60°C and 120°C according to the specific application.

On the other hand, it was also determined that only when Low Viscosity Components 1-3 are used as a low viscosity component (Examples 1-3) is it possible to reduce the traction coefficient which affects fuel economy. This is the first time that it has been determined that the traction coefficient of a composition can be reduced by setting the density of the low viscosity component, in a system which combines a low viscosity component, high viscosity component and control component.

C L A I M S

1. A drive system transmission lubricant oil composition with a kinematic viscosity of from 3.5 to 7.0 mm2/s at 100°C produced by mixing the following:

A. A lubricant base oil having a viscosity of from 1.5 to 6.0 mm^2/s at 15°C and a density of from 0.750 to 0.830 g/cm^3 at 100°C containing at least one of the following: mineral oil, synthetic oil or GTL, as a low viscosity component;

B. from 3 to 35% by weight polyalkylene glycol (PAG) featuring an oxygen/carbon ratio of from 0.450 to 0.580 by weight, as a high viscosity component; and

C. from 1 to 30% by weight of a compound featuring an oxygen/carbon ratio of from 0.080 to 0.350 by weight, as a control component.

2. A drive system transmission lubricant oil composition as claimed in Claim 1 in which the %Cp of the aforementioned low viscosity component ranges from 80 to 100.

3. A drive system transmission lubricant oil composition as claimed in Claim 1 or Claim 2 which exists in a two-phase state when below its separation temperature and exists in a single-phase state when its separation temperature is exceeded, wherein the aforementioned separation temperature ranges from 60°C to 120°C.

4. A drive system transmission lubricant oil composition as claimed in any one of Claims 1 to 3 in which the aforementioned control component is an aliphatic ester featuring a carbon chain having from 4 to 18 carbon atoms excluding the ester group.

5. A drive system transmission lubricant oil composition as claimed in any one of Claims 1 to 4 in which the density of the aforementioned high viscosity component at 20°C ranges from 1.000 to 1.050 g/cm^3.

6. A drive system transmission lubricant oil composition as claimed in any one of Claims 1-5 in which the density of the aforementioned control component at 20°C ranges from 0.800 to 1.000 g/cm^3.

7. A drive system transmission lubricant oil composition as claimed in any one of Claims 1 to 6 in which the traction coefficient at 100°C is 0.0085 or less.

8. A drive system transmission lubricant oil composition as claimed in any one of Claims 1 to 7 which can be used in the lubrication of a drive transmission system for use in automobiles, industrial applications, construction machinery or agricultural machinery.

9. A drive system transmission lubricant oil composition as claimed in any one of Claims 1 to 8 which can be used in a drive system final reduction gear unit.

Figure 1

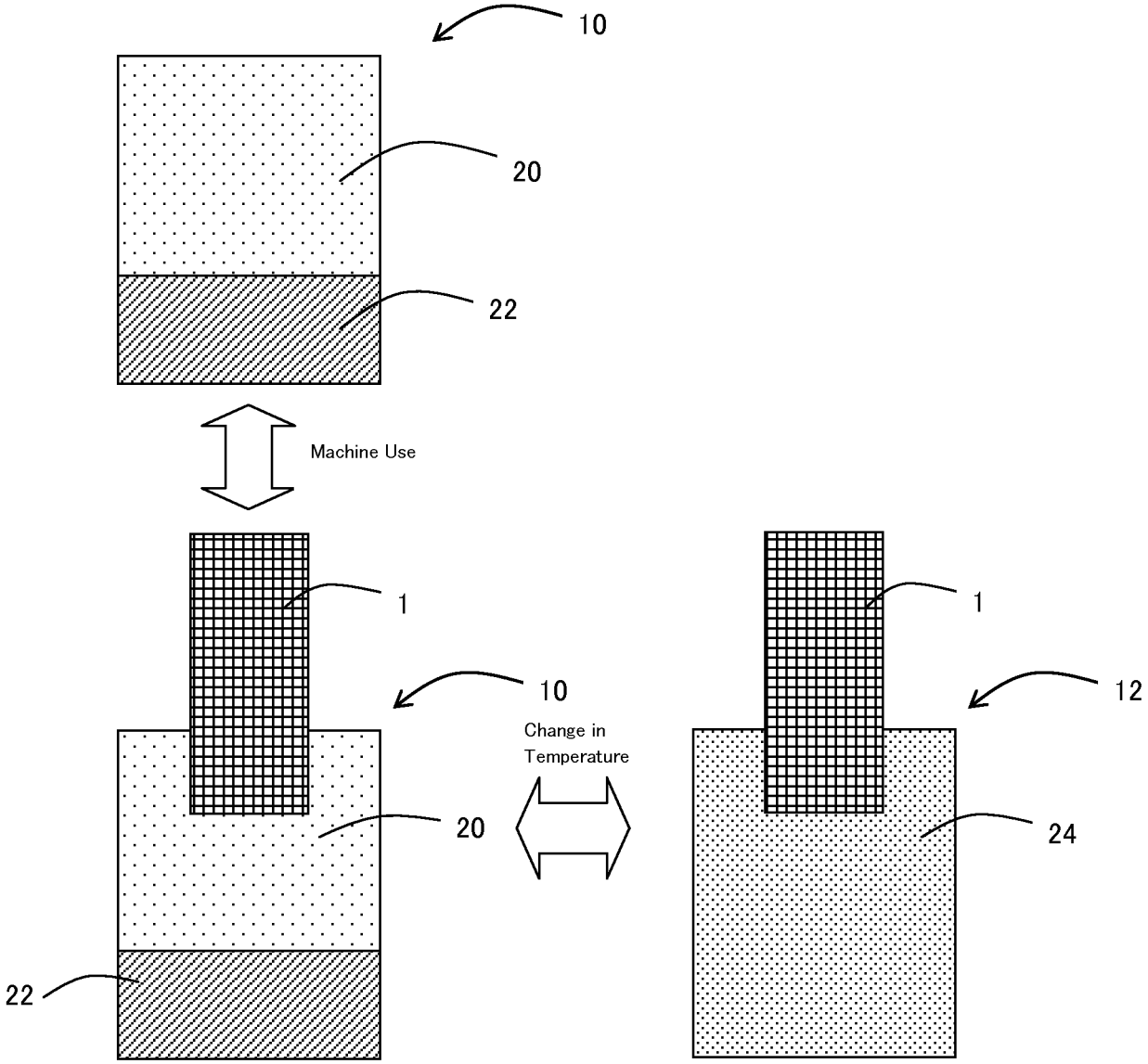
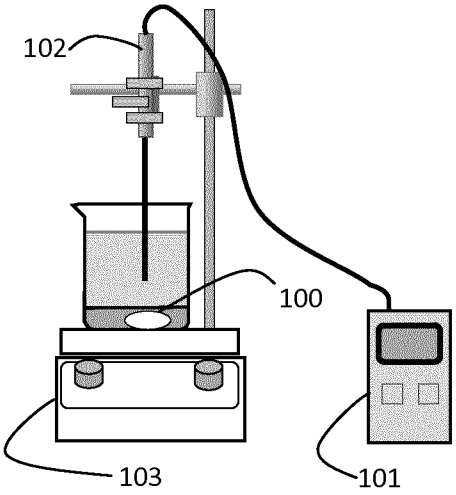


Figure 2



INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2014/063635

A. CLASSIFICATION OF SUBJECT MATTER
INV. C10M169/04 C10M111/04
ADD. C10N20/02 C10N30/02 C10N30/06 C10N40/25

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C10M C10N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2013/010851 A1 (SHELL INT RESEARCH [NL]; KAMATA KUMIKO [JP]; MARUYAMA RYUJI [JP]; SHIN) 24 January 2013 (2013-01-24) page 16, line 28 - page 17, line 11; examples 1-1,1-2,1-6,1-7,29,2-10,2-11,2-12	1-9
A	US 2006/263604 A1 (MARTIN JEAN M [FR] ET AL MARTIN JEAN MICHEL [FR] ET AL) 23 November 2006 (2006-11-23) claim 13	1-9

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
5 September 2014	22/09/2014

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Bertrand, Samuel

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2014/063635

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WO 2013010851 A1	24-01-2013	CN 103703112 A	02-04-2014
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