



(11)

EP 3 256 552 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:

31.07.2019 Bulletin 2019/31

(51) Int Cl.:

C10M 169/04 ^(2006.01) **C10M 169/02** ^(2006.01)

(21) Application number: **16703553.4**

(86) International application number:

PCT/EP2016/052733

(22) Date of filing: **09.02.2016**

(87) International publication number:

WO 2016/128403 (18.08.2016 Gazette 2016/33)

(54) **GREASE COMPOSITION**

SCHMIERFETTZUSAMMENSETZUNG

COMPOSITION DE GRAISSE

(84) Designated Contracting States:

**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR**

(72) Inventor: **DAEGLING, Stefan**

21107 Hamburg (DE)

(30) Priority: **11.02.2015 EP 15154756**

(74) Representative: **Shell Legal Services IP**

p/a Carel van Bylandtlaan 16

2596 HR Den Haag (NL)

(43) Date of publication of application:

20.12.2017 Bulletin 2017/51

(56) References cited:

US-A- 2 862 884 US-A- 4 879 054

US-A1- 2009 088 353 US-A1- 2012 149 613

(73) Proprietor: **Shell Internationale Research**

Maatschappij B.V.

2596 HR The Hague (NL)

EP 3 256 552 B1

Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

DescriptionTechnical Field of the Invention

5 **[0001]** This invention relates to a grease composition and to use of the grease composition in a sealed bearing.

Background of the Invention

10 **[0002]** Sealed bearings are used in many different types of machinery. The seals prevent the ingress of dirt and water that could damage the bearing and impair the functioning of the machinery. Typical seal materials include acrylonitrile-butadiene rubber (NBR) and fluoroelastomers (FKM). A grease is provided at the seal contact in order to prolong the life of the seal. There can be significant friction at the seal-metal interface which can lead to heat generation and a loss of efficiency. The present inventors have sought to provide grease compositions that can act to reduce the friction at the seal-metal interface and thereby improve the efficiency of sealed bearings. US2009/0088353 discloses a grease
15 composition for sealed bearings comprising a Fischer-Tropsch derived base oil and a lithium hydroxystearate thickener.

Summary of the Invention

20 **[0003]** The present inventors have surprisingly found that the combination of a low viscosity polyalphaolefin base oil with a calcium thickener can provide a grease generating particularly low friction at a seal-metal interface. Accordingly the present invention provides a grease composition comprising from 40 to 90wt% of a first base oil component which is a polyalphaolefin and having a kinematic viscosity at 100°C of between 1 and 5mm²/s, wherein the weight percentage is based upon the weight of the grease composition, and comprising 2 to 18 wt % based upon the weight of the grease composition of a thickener which is a C₁₃-C₂₁ fatty acid salt of calcium.

25 **[0004]** In a further aspect the present invention provides the use of a grease composition at a seal-metal interface in a sealed bearing wherein the grease composition comprises a base oil and a thickener, wherein the base oil comprises from 40 to 90wt% of a first base oil which is a polyalphaolefin and has a kinematic viscosity at 100°C of between 1 and 5mm²/s, wherein the weight percentage is based upon the weight of the grease composition, and comprises 2 to 18 wt% based upon the weight of the grease composition of a thickener which is a C₁₃-C₂₁ fatty acid salt of calcium.
30

Detailed Description of the Invention

35 **[0005]** The grease composition comprises from 40 to 90wt% of a first base oil component which is a polyalphaolefin and has a kinematic viscosity at 100°C of between 1 and 5mm²/s. The grease composition may comprise further base oils, but preferably the first base oil component makes up at least 50wt%, and preferably at least 80wt% of the total base oil (based upon the total weight of base oil in the grease composition).

[0006] The thickener in the grease composition is a C₁₃-C₂₁ fatty acid salt of calcium, and preferably is a hydrogenated castor oil fatty acid salt of calcium. The amount of thickener is from 2 to 18wt%, based upon the weight of the grease composition, more preferably from 2 to 14wt%.

40 **[0007]** The grease composition of this invention may also comprise anti-oxidants, rust preventatives, oiliness agents, extreme pressure additives, anti-wear agents, solid lubricants, metal deactivators, polymers, detergents and other additives.

[0008] The anti-oxidants include, for example, 2,6-di-t-butyl-4-methylphenol, 2,6-di-t-butyl-paracresol, P,P'-dioctyldiphenylamine, N-phenyl- α -naphthylamine and phenothiazines.

45 **[0009]** The rust preventatives include paraffin oxide, carboxylic acid metal salts, sulphonic acid metal salts, carboxylic acid esters, sulphonic acid esters, succinic acid esters, sorbitan esters and various amine salts.

[0010] The oiliness agents, extreme pressure additives and anti-wear agents include, for example, sulphurised zinc dialkyl dithiophosphates, sulphurised zinc diaryl dithiophosphates, sulphurised zinc dialkyl dithiocarbamates, sulphurised zinc diaryl dithiocarbamates, sulphurised molybdenum dialkyl dithiophosphates, sulphurised molybdenum diaryl dithiophosphates, sulphurised molybdenum dialkyl dithiocarbamates, sulphurised molybdenum diaryl dithiocarbamates, organic molybdenum complexes, sulphurised olefins, triphenylphosphates, triphenylphosphorothionates, tricresylphosphates, other phosphate esters and sulphurised fats and oils.

[0011] The solid lubricants include, for example, molybdenum disulphide, graphite, boron nitride, melamine cyanurate, PTFE (polytetrafluoroethylene), tungsten disulphide, mica, graphite fluoride and so on.

55 **[0012]** The metal deactivators include, for example, N,N'-disalicylidene-1,2-diaminopropane, benzotriazole, benzimidazole, benzothiazole and thiadiazole.

[0013] The grease compositions may be manufactured using standard grease manufacturing routes.

[0014] The grease compositions of the invention are suitably used at a seal-metal interface in a sealed bearing. The

seal is suitably made from a material such as acrylonitrile-butadiene rubber (NBR), hydrotreated acrylonitrile-butadiene rubber (HNBR) or a fluoroelastomer (FKM). The bearing will contain a lubricant and the grease composition of the invention should be applied such that the grease composition is not contaminated by the lubricant.

Examples

[0015] The invention is further explained in detail below by means of examples and comparative examples, but the invention is in no way limited by these examples.

[0016] Greases were prepared by adding base oil and fatty acid to a kettle, heating to 85-90°C (at 3°C/min) and stirring at 100rpm. Calcium hydroxide was added as a powder and then water was added. The kettle was closed and heated to 135°C (at 2°C/min). The contents were stirred at 100rpm and the wall temperature limit of the kettle was set at 140°C. The kettle was vented approximately 40 minutes after reaching 135°C. Additional base oil was heated to 80°C and then added via a pump to the kettle. The contents were stirred at 150rpm for 5 minutes and the product temperature was kept between 130 and 135°C. A nitrogen tube was connected the kettle, and the contents were exposed to nitrogen flow for 15 minutes, thereby dehydrating the sample. The contents were cooled to 100°C (at 2°C/min) with stirring at 100rpm. Additives were added, using additional base oil for dilution, at 80°C or lower. After addition of an additive, the contents were stirred for 1 minute at 100rpm. The contents were then cooled to 50-60°C (controlled by wall temperature setting: 20°C at 5°C/min and stirring at 100rpm). De-aeration was begun at 70°C, using a vacuum of -150mbar for 10 minutes and stirring at 50rpm. The product was homogenised at 1 x 300bar.

[0017] All the formulations are summarised in Table 1 below, with the quantities given as wt% based upon the total weight of the formulation:

Table 1

		Example 1	Comparative Example 1	Comparative Example 2
Base Oil	PAO 2.0	78.46	-	-
	XHVI 3.0	-	78.46	-
	XHVI 4.0	-	-	78.46
Thickener	CaOH	1.64	1.64	1.64
	HCOFA	12.50	12.50	12.50
Additive Package		7.40	7.40	7.40

[0018] The PAO 2.0 was a polyalphaolefin and had a viscosity at 100°C of 2.0cSt. The XHVI 3.0 and XHVI 4.0 were base oils made by a Fischer-Tropsch process and were obtained from Shell. The XHVI 3.0 had a viscosity at 100°C of 3.0cSt and the XHVI 4.0 had a viscosity at 100°C of 4.0cSt. HCOFA is hydrogenated castor oil fatty acid. The additive package was the same in each of the example and comparative examples.

[0019] The greases were applied to NBR seals. A sealed bearing was run for 24 hours and then the friction was measured.

[0020] The results are given in table 2:

Table 2

	Friction level (Ncm)
Example 1	1.88
Comparative Example 1	3.06
Comparative Example 2	3.12

[0021] The grease composition of the invention (wherein the base oil is a polyalphaolefin) shows considerably lower friction than the grease compositions of the comparative examples (wherein the base oils have similar viscosities but are made by Fischer-Tropsch processes).

Claims

1. A grease composition comprising from 40 to 90wt% of a first base oil component which is a polyalphaolefin and having a kinematic viscosity at 100°C of between 1 and 5 mm²/s, wherein the weight percentage is based upon the weight of the grease composition, and comprising a thickener which is a C₁₃-C₂₁ fatty acid salt of calcium, wherein the amount of thickener is from 2 to 18wt%, based upon the weight of the grease composition,
2. Use of a grease composition at a seal-metal interface in a sealed bearing wherein the grease composition comprises a base oil and a thickener, wherein the base oil comprises from 40 to 90wt% of a first base oil component which is a polyalphaolefin and has a kinematic viscosity at 100°C of between 1 and 5mm²/s, wherein the weight percentage is based upon the weight of the grease composition, and comprises a thickener which is a C₁₃-C₂₁ fatty acid salt of calcium, wherein the amount of thickener is from 2 to 18wt%, based upon the weight of the grease composition,

Patentansprüche

1. Schmierfettzusammensetzung, umfassend 40 bis 90 Gew.-% einer ersten Grundölkomponte, bei der es sich um ein Polyalphaolefin handelt und deren kinematische Viskosität bei 100 °C zwischen 1 und 5 mm²/s beträgt, wobei der Gewichtsprozentsatz auf das Gewicht der Schmierfettzusammensetzung bezogen ist, und umfassend ein Verdickungsmittel, bei dem es sich um ein C₁₃-C₂₁-Calciumfettsäuresalz handelt, wobei die Menge an Verdickungsmittel bezogen auf das Gewicht der Schmierfettzusammensetzung 2 bis 18 Gew.-% beträgt.
2. Verwendung einer Schmierfettzusammensetzung an einer Dichtung-Metall-Grenzfläche in einem abgedichteten Lager, wobei die Schmierfettzusammensetzung ein Grundöl und ein Verdickungsmittel umfasst, wobei das Grundöl 40 bis 90 Gew.-% einer ersten Grundölkomponte umfasst, bei der es sich um ein Polyalphaolefin handelt und deren kinematische Viskosität bei 100 °C zwischen 1 und 5 mm²/s beträgt, wobei der Gewichtsprozentsatz auf das Gewicht der Schmierfettzusammensetzung bezogen ist, und ein Verdickungsmittel umfasst, bei dem es sich um ein C₁₃-C₂₁-Calciumfettsäuresalz handelt, wobei die Menge an Verdickungsmittel bezogen auf das Gewicht der Schmierfettzusammensetzung 2 bis 18 Gew.-% beträgt.

Revendications

1. Composition de graisse comprenant de 40 à 90 % d'un premier composant d'huile de base qui est une polyalphaoléfine et ayant une viscosité cinématique à 100°C comprise entre 1 et 5 mm²/s, dans laquelle le pourcentage massique est basé sur la masse de la graisse, et comprend un épaississant qui est un sel calcique d'acide gras en C₁₃ à C₂₁, dans laquelle la quantité d'épaississant va de 2 à 18 %, sur la base de la masse de la composition de graisse.
2. Utilisation d'une composition de graisse au niveau d'une interface joint-métal dans un roulement étanche, dans laquelle la composition de graisse comprend une huile de base et un épaississant, l'huile de base comprenant de 40 à 90 % d'un premier composant d'huile de base qui est une polyalphaoléfine ayant une viscosité cinématique à 100°C comprise entre 1 et 5 mm²/s, dans laquelle le pourcentage massique est basé sur la masse de la composition de la graisse, et comprend un épaississant qui est un sel calcique d'acide gras en C₁₃ à C₂₁, dans laquelle la quantité de l'épaississant est de 2 à 18 %, sur la base de la masse de la composition de graisse.

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- US 20090088353 A [0002]