



US 20030144159A1

(19) **United States**

(12) **Patent Application Publication**

Akiyama et al.

(10) **Pub. No.: US 2003/0144159 A1**

(43) **Pub. Date: Jul. 31, 2003**

(54) **FLUORESCENT GREASE AND BEARINGS
HAVING THE SAME THEREIN**

(30) **Foreign Application Priority Data**

Jun. 8, 2000 (JP) 2000-171823

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Publication Classification

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(51) **Int. Cl.⁷** **C10M 169/06**

(52) **U.S. Cl.** **508/462; 508/247; 508/539**

(21) Appl. No.: **10/265,309**

(57) **ABSTRACT**

(22) Filed: **Sep. 26, 2002**

Related U.S. Application Data

(63) Continuation-in-part of application No. 09/755,318,
filed on Jan. 5, 2001, now abandoned.

The invention provides a fluorescent grease which enables monitoring of behaviors of the grease for elucidation of the mechanism of grease lubrication, and a rolling element bearing or a condition monitoring rolling element bearing, both charged with the fluorescent grease. The fluorescent grease contains, as its essential components, a base oil, a thickener agent, and a fluorescent substance.

FIG. 1

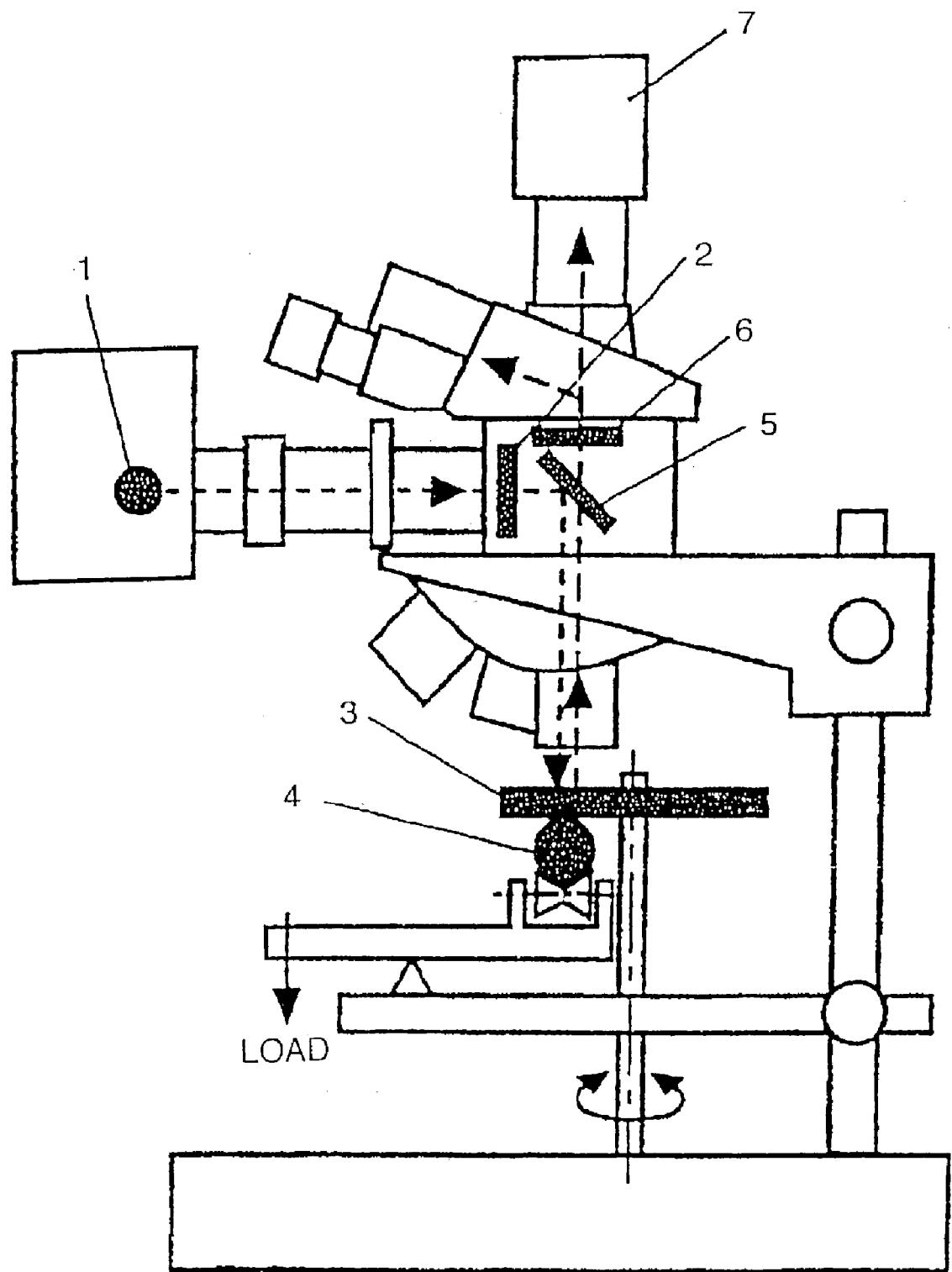


FIG. 2 (A)

HERTZIAN STATIC
CONTACT

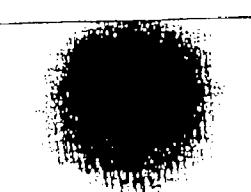


FIG. 2 (B)

UNI-DIRECTIONAL
SLIDING CONTACT
AT FIRST CYCLE



FIG. 2 (C)

UNI-DIRECTIONAL
SLIDING CONTACT
AT 10TH CYCLE

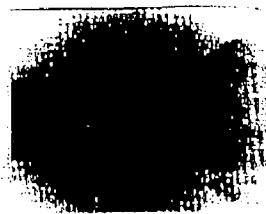


FIG. 2 (D)

SLIDING CONTACT
AT THE MIDDLE OF
FIRST CYCLE IN
RECIPROCATING TEST

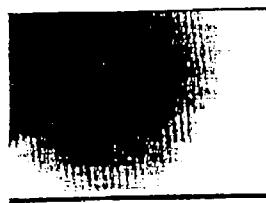


FIG. 2 (E)

SLIDING CONTACT
DURING
DECELERATION IN
FIRST CYCLE IN
RECIPROCATING TEST

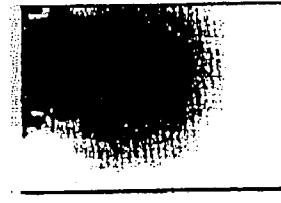


FIG. 2 (F)

SLIDING CONTACT
JUST AFTER THE
REVERSAL OF MOTION
IN FIRST CYCLE IN
RECIPROCATING TEST

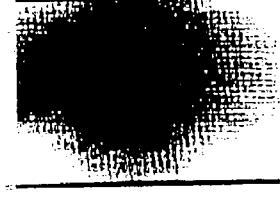


FIG. 2 (G)

SLIDING CONTACT AT
THE MIDDLE OF 10TH
CYCLE IN
RECIPROCATING TEST



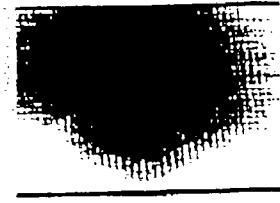
FIG. 2 (H)

SLIDING CONTACT
DURING
DECELERATION IN
10TH CYCLE IN
RECIPROCATING TEST



FIG. 2 (I)

SLIDING CONTACT
JUST AFTER THE
REVERSAL OF MOTION
IN 10TH CYCLE IN
RECIPROCATING TEST



200µm

FLUORESCENT GREASE AND BEARINGS HAVING THE SAME THEREIN

BACKGROUND OF THE INVENTION

[0001] 1. Field of the Invention

[0002] This invention relates to a grease, which is able to realize a low torque of a rolling element bearing and improve wear resistance as a result of visual observation of a grease lubricant film wherein the formation of the grease lubricant film and the behavior of its break-down that have never been satisfactorily elucidated up to now, i.e. the actions of a base oil and a thickener agent under non-steady state operating conditions, are made clear. The invention also relates to a bearing or condition monitoring rolling element bearing charged with such a grease as mentioned above.

[0003] 2. Description of the Prior Art

[0004] For the observation or measurement of a lubricant film, there have been used a number of methods including, aside from a fluorescence method, a light interference method, a micro displacement transducer method, an X-ray transmission method, an electric capacitance method, an electric resistance method, a method using an ultrasonic wave, and the like. Of these observation or measurement methods, the most widely employed method of directly measuring a film thickness is the light interference method or its extension of ultra thin film interferometry.

[0005] For the indirect measurement of a film thickness of the order of size of molecules, the micro displacement transducer method has been used. However, with the light interference method, it is necessary that a specific type of coating film be formed on a test piece. On the other hand, the micro displacement transducer method is disadvantageous in that this method is applicable only to conditions where the elastic deformation of sliding surfaces is negligible.

[0006] Moreover, it is not only oil but greases that are used as a lubricant in rolling element bearings. The fluorescence method has been applied only in the studies of oil lubrication, and the use of a fluorescent grease as a grease in these methods has never been known.

SUMMARY OF THE INVENTION

[0007] It is accordingly an object of the invention to provide a fluorescent grease which enables monitoring of behaviors of a grease for elucidation of the mechanism of grease lubrication.

[0008] It is another object of the invention to provide a rolling element bearing or condition monitoring rolling element bearing charged with the fluorescent grease.

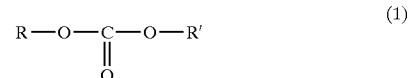
[0009] Based on the very simple principle that when a proper amount of a fluorescent substance exists, it can be observed as it shines bright, fluorescent substances have been applied to many aspects of tribology. The best known application is directed to the measurement of an oil film thickness between a piston ring and a cylinder liner of an automotive vehicle, for which a measuring method using a laser induced fluorescence technique has been established in recent years. Fluorescent substances have also been applied to studies of oil flow between an oil seal and a rotary shaft or spindle. With an ordinary light interference method, a film thickness can be measured down to a level as small as about

$\frac{1}{4}$ of the wavelength of visible light, i.e. about 100 nm. In contrast, when using the fluorescence method, the limitation of the measurement can be decreased to the order of nanometers provided that the fluorescent intensity and measuring sensitivity can be increased.

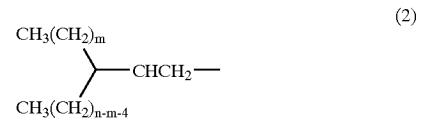
[0010] The lubricating mechanism of a grease is more complicated than that of ordinary oil lubrication because the grease consists of a thickener agent and a base oil, and thus, any theory of accurately predicting a lubricant film thickness for a given set of operating conditions has not been established yet.

[0011] According to the invention, there is provided a fluorescent grease which comprises a base oil dissolving a fluorescent substance therein, and a thickener agent added to the base oil.

[0012] The base oil used is a synthetic carbonate ester of the following general formula (1)



[0013] wherein R and R' may be the same or different and independently represent a branched alkyl group having from 13 to 15 carbon atoms. More specifically, in the general formula (1), R and R' may be the same or different and independently represent a branched alkyl group of the following general formula (2)



[0014] wherein n=13 to 15 and m=0 to 6.

[0015] There are listed, as the thickener agents, an alkali metal salt and/or an alkaline earth metal salt, which is prepared from an alkali metal or alkaline earth metal hydroxide and a higher fatty acid having 10 or more carbon atoms or a higher hydroxy-fatty acid having one or more hydroxyl group and 10 or more carbon atoms. Typical examples include lithium soaps such as lithium stearate, lithium 12-hydroxystearate, and the like.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016] FIG. 1 is a schematic drawing of a fluorescence microscope used in the Examples of the invention; and

[0017] FIGS. 2(A) to 2(I) are, respectively, fluorescence images of a lubricant film of a fluorescent grease according to the invention.

PREFERRED EMBODIMENTS OF THE INVENTION

[0018] In the practice of the invention, any fluorescent substances, which are likely to be dissolved in a base oil, may be usable.

[0019] Examples of such substances include condensed ring-type hydrocarbons such as pyrene, perylene, anthracene, naphthacene, chrysene, triphenylene, pentaphene, 1,2-benzpyrene, hexaphene, triaphthylene, coronene, tetracene, dibenzcoronene, rubrene and the like, diphenylpolyenes such as p-diphenylbenzene (p-t-phenyl), 1,6-diphenyl-1,3,5-hexatriene, 1,8-diphenyl-1,3,5,7-octatetraene and the like, polycyclic hydrocarbons such as tetraphenyl-p-quinodimethane, asy-phenyl-o-tolyl-p-quinodimethane, p-distyrylbenzene and the like, benzene derivatives such as hydroquinone dimethyl ether, polycyclic compounds such as derivatives of benzidine, stilbene and the like, condensed ring-type compounds such as naphthylamine and derivatives thereof, and heterocyclic derivatives such as thiadiazole derivatives, pyridine derivatives, benzoxazole derivatives, flavone derivatives, coumarin derivatives, quinoline derivatives, acridine derivatives and the like.

[0020] Moreover, dyes may be used in combination, including fluorescein, acridine orange, methylene blue, rosamine, trypaflavin, thionine, pyronin G, resorcinol, eosin, erythrosin, rhodamine B, chlorophyll a, chlorophyll b, benzoflavin, phenosafranine and the like.

[0021] Preferred fluorescent substances include one or more of those substances selected from pyrene, perylene, 1,6-diphenyl-1,3,5-hexatriene, 1,8-diphenyl-1,3,5,7-octatetraene, and coumarine 6.

[0022] Most preferably, mention is made of pyrene, which exhibits the highest fluorescent intensity with little bleaching phenomenon (attenuation of fluorescent intensity) being recognized when a CCD camera and a high pressure mercury lamp as a light source are employed. The saturation concentration in a mineral hydrocarbon, PAO or a carbonate is at a level of about 2 mass %.

[0023] Pyrene has a peak wavelength of absorbed light at 336 nm and a peak wavelength of eximer emission at 480 nm (when dissolved in cyclohexane). Accordingly, bluish green fluorescence is emitted on irradiation of UV light in the vicinity of the peak wavelength. It will be noted that the absorption wavelength depends on the type of fluorescent substance, and perylene has an absorption peak in the visible light region, so that fluorescence can be observed without use of any specific type of light source such as a mercury lamp.

[0024] The grease of the invention may further comprise additives ordinarily employed in greases, such as an antioxidant, a rust inhibitor, an extreme pressure agent and the like.

[0025] The embodiments of the invention may be summarized as follows.

[0026] (1) A fluorescent grease, which comprises, as essential components, a base oil, a thickener agent, and a fluorescent substance.

[0027] (2) A fluorescent grease as recited in (1) above, wherein the fluorescent substance is at least one member selected from pyrene, perylene, 1,6-diphenyl-1,3,5-hexatriene, 1,8-diphenyl-1,3,5,7-octatetraene, and coumarine 6.

[0028] (3) A fluorescent grease as recited in (1) or (2) above, wherein the base oil consists of a synthetic carbonate ester, and the thickener agent consists of a lithium soap.

[0029] (4) A rolling element bearing having a fluorescent grease recited in any one of (1) to (3) above filled therein.

[0030] (5) A condition monitoring rolling element bearing having a fluorescent grease recited in any one of (1) to (3) above filled therein.

[0031] (6) In another embodiment of the present invention, the fluorescent grease contains at least two different fluorescent substances. The fluorescent grease is then charged to the rolling element bearing or condition monitoring rolling element bearing and the bearing irradiated with light of multiple wavelengths. The bearing is then rotated and the behavior of the fluorescent grease observed by a CCD camera. Plural images of the fluorescent substances appearing on the CCD are then compared with each other to help determine the characteristics of the grease.

[0032] The invention is more particularly described by way of example.

[0033] FIG. 1 shows a fluorescence microscope for observing the behavior of a grease lubricant film between a steel ball and a flat glass disk by use of fluorescent grease.

[0034] A fluorescence microscope has an exciter filter 2 for passing a light ray with a wavelength band capable of exciting a fluorescent substance from light rays emitted from a light source 1 (mercury lamp), a barrier filter 6 and a dichroic mirror 5 capable of cutting reflected light and passing a fluorescence alone, a glass disk 3, a ball (steel ball) 4, and a CCD camera 7.

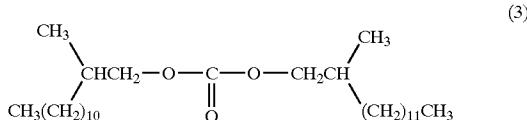
[0035] The fluorescent wavelength of a fluorescent substance generally has a wavelength longer than an absorption wavelength, so that a cutoff wavelength or a transmitted wavelength band is selected by means of the filters so as to allow irradiation of light of a shorter wavelength and observation of light of a longer wavelength.

[0036] With pyrene, there were used the exciter filter 2 with a transmitted wavelength band of 330 to 380 nm, and the dichroic mirror 5 and the barrier filter 6 having cut-off wavelengths of 400 nm and 420 nm, respectively.

[0037] A testing device was so designed that the contact portion of the glass disk and the steel ball was provided below an objective and a fluorescence of a lubricant film was taken with the CCD camera. The glass disk was made of a non-fluorescent synthetic quartz glass having a thickness of 3 to 8 mm, and its drive shaft was driven with an AC servo motor via a timing belt. The ball (steel ball) 4 was freely supported by means of two tapered rollers, and was in rolling contact with the glass disk 3 by traction with the glass disk 3. The ball 4 could also be rigidly supported to a loading arm so as to afford pure sliding contact between the ball and the glass disk 3. The motor could permit several driving modes such as of a constant speed motion, acceleration and deceleration, reciprocating motion and the like.

EXAMPLE 1

[0038] A grease was prepared and had a formulation of 80 mass % of a synthetic carbonate ester of the following chemical formula used as a base oil, and 20 mass % of a lithium stearate soap used as a thickener agent.



[0039] The base oil had a viscosity of 18 mm²/s (40° C.), and the grease had a worked consistency of 198 (25° C.) and a dropping point of 197° C.

[0040] The thus prepared grease was applied between the steel ball and the glass disk, each cleaned with heptane, followed by uni-directional and reciprocating sliding contact to observe the behavior thereof.

[0041] The load applied was 4.9 N, and the speed was set at 10 levels ranging from 23 mm/s to 230 mm/s for the uni-directional contact and also at 10 levels ranging from 23 mm/s to 230 mm/s for the reciprocating contact. The reciprocating contact was performed for 20 reciprocating cycles wherein an amplitude was 34.5 mm and an acceleration was 3.46 m/s² at the time of acceleration or deceleration. When the test was performed, the room temperature was at 15° C.

[0042] The fluorescence images of the lubricant films of the greases are shown in FIGS. 2(A) to 2(I).

[0043] FIG. 2(A) shows a fluorescence image of a grease at a standstill. The contact area at the center is dark, and it is brighter outside the contact. The brightness is proportional to the amount of fluorescent substance, from which it will be seen that the gap between the ball and the glass disk increases with the distance from the contact center.

[0044] FIG. 2(B) shows a fluorescence image in the uni-directional motion. A dark portion is observed at the outlet of the contact and is considered as a cavity created through the formation of a hydrodynamic lubricating film.

[0045] FIG. 2(C) shows a fluorescence image at the tenth cycle of the uni-directional motion. Something like a cavity starts to appear near the inlet of the contact. This is considered to be an air cavity formed in the previous cycles remaining within the soap fibers overrolled on the track, or a cavity formed by hydrodynamic action at a dam of the grease piled up at the inlet and side of the contact.

[0046] FIGS. 2(D), 2(E) and 2(F), respectively, show fluorescence images at the first cycle of the reciprocating test wherein FIG. 2(D) is taken at the middle of the reciprocating stroke, FIG. 2(E) is taken during deceleration, and FIG. 2(F) is taken just after the reversal of motion.

[0047] It will be seen that at the reversal of motion, the re-supply of the base oil is insufficient to cause the base oil starvation at the inlet.

[0048] FIGS. 2(G), 2(H) and 2(I), respectively, show fluorescence images at the tenth cycle of the reciprocating test wherein FIG. 2(G) is taken at the middle of the reciprocating stroke, FIG. 2(H) is taken during deceleration, and FIG. 2(I) is taken just after the reversal of motion. Comparison of these images reveals that the inlet starvation of the base oil becomes more significant at the tenth cycle than at the first cycle. More particularly, it will be noted that in the grease lubrication, the base oil is squeezed out from

the track, and the re-supply of the base oil onto the track is retarded, especially after the reversal of motion.

EXAMPLE 2

[0049] The grease characteristics have been tested using a device shown in FIG. 1 by adding "rhodamine B" to the fluorescent grease consisting of the base oil, the thickener agent and the fluorescent substance which is the same as that shown in Example 1.

[0050] The fluorescent grease was irradiated with light with wavelengths of 340 nm and light with wavelengths of 550 nm, on which light the fluorescent substance reacts, and the fluorescent grease was observed by a CCD camera. An image of the fluorescent substance irradiated with the light with wavelengths of 340 nm and that irradiated with the light with wavelengths of 550 nm were substantially the same, and confirmed that the base oil and the thickener agent coexisted with each other and did not separate from each other. Further, fluorescent grease was irradiated with light with a wavelength of 340 nm and light with a wavelength of 550 nm, and the fluorescent grease was observed by a CCD camera using "calcium stearate" instead of the thickener agent. Images of the fluorescent substance irradiated with the light with a wavelength of 340 nm and that irradiated with the light with a wavelength of 550 nm were different from each other, and revealed that the base oil and the thickener were separated from each other.

EXAMPLE 3

[0051] Fluorescent grease comprising the base oil and the thickener agent, which is the same as that used in Example 1, was tested by an acceleration testing device, and it deteriorated.

[0052] The fluorescent grease was tested for 100 hours, 500 hours and 1,000 hours and respectively collected, and the fluorescent substance and "rhodamine B" was added to the fluorescent grease in the same manner as Example 2, and the grease characteristics were tested by the device shown in FIG. 1 wherein an image of the fluorescent substance irradiated with the light with a wavelength of 340 nm was compared with that irradiated with the light with a wavelength of 550 nm. As time passed, the base oil separated from the thickener agent.

EXAMPLE 4

[0053] 30% of a bearing space of a mechanically sealed bearing was charged with the fluorescent grease comprising the base oil, the thickener agent and the fluorescent substance, then the same bearing was rotated at 140° C. for 100 hours at 20,000 rpm, and fluorescent grease dispersed from the bearing was received by a test piece (paper) while the circumference of the bearing was surrounded by the test piece.

[0054] When the test piece was irradiated with light and a grease spot on the test piece was examined, it was revealed that many fine spots were distributed.

[0055] Further, 30% of a bearing space of a mechanically sealed bearing was charged with the fluorescent grease comprising the base oil, the thickener agent and the fluorescent substance, then the same bearing was rotated at 100° C. for 100 hours at 20,000 rpm and fluorescent grease

dispersed from the bearing was received by a test piece (paper) while the circumfluence of the bearing was surrounded by the test piece.

[0056] When the test piece was irradiated with light and a grease spot on the test piece was examined, fine spots were hardly found.

[0057] Still further, 10% of a bearing space of a mechanically sealed bearing was charged with the fluorescent grease comprising the base oil, the thickener agent and the fluorescent substance, then the same bearing was rotated at 100° C. for 100 hours at 20,000 rpm and fluorescent grease dispersed from the bearing was received by a test piece (paper) while the circumfluence of the bearing was surrounded by the test piece.

[0058] When the test piece was irradiated with light and a grease spot on the test piece was examined, fine spots were hardly found.

[0059] It was verified by the test that the bearing charged with the fluorescent grease, which was charged in the bearing space by 30% or 10%, was durable to a rotation of 20,000 rpm in the case of the testing temperature of 100° C.

[0060] It has generally been considered that ability of the grease to re-supply the base oil onto the overrolled track influences the degree of oil starvation at the contact. As will be apparent from the above, this can be confirmed by the direct observation of behaviors of the fluorescent grease of the invention. This means that when a fluorescent grease is charged to a bearing, condition monitoring becomes possible.

[0061] Analysis of the intensity of fluorescence enables one to quantitatively determine an amount of a residual base oil and an amount of re-supplied or a returned base oil.

[0062] In the reciprocating sliding contact, it has been appreciably confirmed that the inlet starvation of a base oil caused by a residual cavity becomes remarkable at the reversal of motion. In order to reduce the traction at the reversal of motion, it will be necessary to develop a proper combination of a thickener agent and a base oil that allows better oil release and anti-foaming properties.

What is claimed:

1. A fluorescent grease comprising, as essential components, a base oil consisting of a synthetic carbonate ester, a thickener agent consisting of a lithium soap and a fluores-

cent substance consisting of at least one member selected from the group consisting of pyrene, perylene, 1,6-diphenyl-1,3,5-hexatriene, 1,8-diphenyl-1,3,5,7-octatetraene and coumarine 6.

2. The fluorescent grease of claim 1, wherein the fluorescent substance is at least one member selected from the group consisting of pyrene, perylene, 1,6-diphenyl-1,3,5-hexatriene and 1,8-diphenyl-1,3,5,7-octatetraene.

3. A rolling element bearing charged with the fluorescent grease of claim 1.

4. A condition monitoring rolling element bearing charged with the fluorescent grease of claim 1.

5. A method of testing grease characteristics using the fluorescent grease of claim 1, comprising the steps of:

adhering the fluorescent grease to a ball;

rotating the ball while pressing the ball onto a glass substrate;

irradiating the ball with light through the glass substrate; and

observing behavior of the fluorescent grease with a CCD camera while the ball is being rotated.

6. The method of claim 5, wherein the fluorescent grease is made of at least two fluorescent substances, the ball is irradiated with light of multiple wavelengths and plural images of the fluorescent substances appearing on the CCD are compared with one another.

7. The method of claim 6, wherein at least one of the fluorescent substances has an affinity for the base oil and another fluorescent substance has an affinity for the thickening agent.

8. A method of testing bearings characteristics comprising the steps of:

charging a bearing with the fluorescent grease of claim 1; rotating the bearing at a specified temperature for a specified time and at a specified speed;

receiving fluorescent grease dispersed from the bearing by a test piece;

irradiating the test piece with light; and

examining a grease spot on the test piece.

9. The method of claim 8, wherein the bearing is a mechanically sealed bearing.

* * * * *