

A method for refining lubricant for use on a magnetic recording medium

## Technical field of the invention

5       The present invention relates to a method for refining lubricant for use on a magnetic recording medium, to lubricant that is refined by the method, and a magnetic recording medium that uses the lubricant.

## 10   Background of the invention

A recording medium is commonly used in a fixed magnetic recording medium device as a data recording medium of a computer. Such a fixed magnetic recording medium device comprises a driving mechanism for a magnetic recording medium, a driving mechanism  
15   for a magnetic head, a stopping mechanism for a magnetic head, a data transfer and control mechanism, and one or more magnetic recording media mounted on the device. Significant tendencies in the development of the fixed magnetic recording medium in recent years are high recording density, large capacity, and high data transfer  
20   speed.

In a conventional fixed magnetic recording medium device, a magnetic head floats when the magnetic recording medium is rotating, and the magnetic head contacts with the surface of the

magnetic recording medium when a driving motor for rotating the medium stops. This operation scheme is called a CSS (contact start and stop) scheme, and is commonly used in a usual recording device. When the magnetic recording medium begins to rotate and stops in this scheme, the head slides and rubs with the surface of the magnetic recording medium. To protect the magnetic layer from this friction, a protective layer is laminated; and to improve surface lubrication performance, a lubricating film is laminated.

Rotating speed of a medium is increasing from about 5,400 rpm (revolutions per minute) to the range between 7,200 to 15,000 rpm, recently. As a result, a spin migration phenomenon is growing significantly, in which the lubricant on the medium surface moves towards outer periphery due to centrifugal force and scatters.

Thickness increase of the lubricant in the peripheral region due to the spin migration causes a problem of sticking. Higher speed of rotation gives rise to heating, which causes evaporation of the lubricant. The evaporation of the lubricant decreases thickness of the lubricant, which may cause wear of the protective layer. A head crash may happen in the worst case.

In order to suppress the spin migration, the lubricant film is necessarily formed with uniform thickness and with stability on the surface of the protective layer, and high adhesiveness and strong

bonding between the lubricant film and the protective layer are important. The evaporation of the lubricant is also prevented by enhancement of the adhesiveness and bonding strength. To enhance the adhesiveness and bonding strength, a perfluoropolyether  
5 lubricant having an end group of hydroxyl group or piperonyl group is used. The examples of such lubricant are “Fomblin Z DOL” and “AM3001”, which are commercially available from Ausimont K. K., Tokyo, Japan. Most of the perfluoropolyether lubricant used at present has a number average molecular weight in the range between  
10 1,000 and 10,000. If the molecular weight is too low, lubricating performance and heat resistance degrades; if the molecular weight is too high, adhesion tends to occur. However, these lubricants deteriorate durability to the spin migration at the high rotating speed in the range between 7,200 to 15,000 rpm, although the adhesiveness  
15 and bonding strength of the lubricant are enough at the rotating speed of about 5,400 rpm that is employed at present.

Accordingly, development is being made for the material having a functional group with strong polarity that is introduced into a  
20 perfluoropolyether lubricant in order to enhance adhesiveness and bonding. Japanese Unexamined Patent Application Publication Nos. H11-172268 and 2001-6155, for example, disclose that introduction of an amine end group significantly enhances bonding strength. Synthesis of these end-group-modified materials, however, requires

long time and dedicated equipment, which arouses apprehension for increased cost for a manufacturer of magnetic recording media to synthesis these materials in the own firm.

5       Commercially available lubricant “Fomblin Z tetraol” from Ausimont K. K., Tokyo, Japan is a perfluoropolyether having a molecular structure in which two hydroxyl groups are introduced at each of the both ends. The lubricant is superior to “Fomblin Z DOL” from the viewpoint of the adhesiveness and bonding strength with the  
10   protective layer. The lubricant is known to contain a coexisting lubricant that has a structure including only one hydroxyl group at the end group. The structure including only one hydroxyl group at the end group is nothing but the structure of “Fomblin Z DOL”. The coexistence of this lubricant component causes deterioration of  
15   adhesiveness and bonding strength from the expected performance.

      Ionic impurities contained in lubricant cause corrosion on a magnetic recording medium. The ionic impurities induce dissolution of cobalt used in a magnetic layer. The cobalt ions act as a catalyst  
20   and dissolve perfluoropolyether lubricant molecules. Therefore, the ionic impurities are to be eliminated as thoroughly as possible. However, since the lubricant inevitably contains ionic impurities that are used in the synthesis process of the perfluoropolyether lubricant, the ionic impurities must be removed before they are applied to a

magnetic recording medium.

Problem to be solved by the invention

In order to suppress spin migration and evaporation of a  
5 lubricant at high rotating speed of 7,200 to 15,000 rpm and at high  
temperature and high humidity of about 80°C and 80% RH, for  
example, the lubricant is required to have high adhesiveness and  
strong bonding with a protective layer. The methods disclosed in  
Japanese Unexamined Patent Application Publication Nos. H11-  
10 172268 and 2001-6155 are insufficient in view of required time and  
costs.

In addition, a perfluoropolyether lubricant contains remaining  
ions that are used in the synthesis process of the perfluoropolyether.  
15 The ionic impurities must be removed before coating on a magnetic  
recording medium.

Accordingly, an object of the present invention is to provide a  
method for refining a perfluoropolyether lubricant that has high  
20 adhesiveness and strong bonding with a protective layer, in order to  
provide a magnetic recording medium that exhibits excellent  
lubricating performance and long term stability even under high  
rotating speed and high temperature environment of the magnetic  
recording medium used in a fixed magnetic recording device.

Another object of the present invention is to provide a lubricant that is given high adhesiveness and strong bonding with a protective layer by means of this refining method. Still another object of the invention is to provide a magnetic recording medium that employs  
5 such a lubricant.

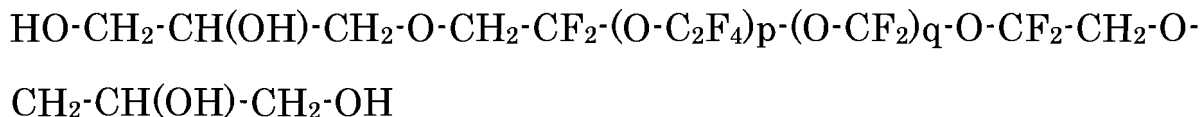
#### Means to solve the problem

The first aspect of the invention is a method for refining a  
10 lubricant that contains at least two types of compounds having different polarity. The method comprises steps of putting the lubricant into a pressure vessel in which pressure and temperature are set at certain values; removing the compound with weaker polarity dissolved in an extracting medium of supercritical carbon  
15 dioxide fluid that is flowing at a specified rate through the pressure vessel held at a specified temperature and pressure; and one of the two steps of recovering the target compound dissolved in the extracting medium that is flowing through the pressure vessel held under a state at the specified pressure and at a temperature lower  
20 than the specified temperature, and recovering the target compound dissolved in the extracting medium that is flowing through the pressure vessel held under a state at the specified temperature and at a pressure higher than the specified pressure. Preferably, the specified temperature is in the range from 31°C to 100°C and the

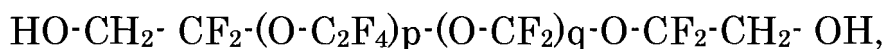
specified pressure is in the range from 7.4 MPa to 30 MPa. More preferably, the specified temperature is in the range from 40°C to 100°C. Most preferably, the removing step is conducted under a state at 60°C and 15 MPa or a state exhibiting equivalent dissolving ability to the state at 60°C and 15 MPa, and the recovering step is conducted under a state at 60°C and 20 MPa or a state exhibiting equivalent dissolving ability to the state at 60°C and 20 MPa.

Advantageously, the method of the invention comprises a step of removing the lubricant B represented by formula (2) and the step of recovering the lubricant A represented by formula (1);

lubricant A, formula (1):



lubricant B, formula (2):



where p and q are positive integers.

The second aspect of the present invention is a lubricant for use on a magnetic recording medium being refined by the above-described method, in which ionic impurities are removed from the lubricant and the lubricant is sorted based on end group structure of the perfluoropolyether molecule of the lubricant. The lubricant is preferably a mixture of the lubricant A having a molecular structure

represented by the formula (1) and the lubricant B having a molecular structure represented by the formula (2). The lubricant preferably includes B in an amount from zero to 10 molar percent with respect to molar amount of the mixture of A and B.

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The third aspect of the present invention is a magnetic recording medium using the refined lubricant as described above.

#### Aspects of embodiments of the invention

10 The present invention will be described in detail in the following.

The first aspect of the invention is a method for refining a lubricant that contains at least a compound having two hydroxyl groups in each of the two end groups of the lubricant molecule and a  
15 compound having one hydroxyl group in each of the two end groups of the lubricant molecule. The method of the first aspect obtains a target lubricant by sorting the lubricant molecules based on polarity of the end groups. The method uses an extracting fluid of the supercritical carbon dioxide and is based on the fact that solubility of  
20 a polar component into the supercritical carbon dioxide fluid is low at a relatively low pressure or at a relatively high temperature.

This method needs a vessel having a feed port and a vent port for the supercritical carbon dioxide fluid and equipment to control

pressure and temperature in the vessel to a desired value, in order to refine the lubricant. The pressure vessel and equipment for use in the refining process include, for example, a thick-wall stainless steel vessel having an inlet and an outlet, a pump for flowing the

5 supercritical carbon dioxide: SCF-Get manufactured by JASCO Corporation, Tokyo, Japan, a full automatic pressure regulator valve: SCF Bpg manufactured by JASCO Corporation, Tokyo, Japan, and a column oven used in usual chromatography. An extracting medium used is supercritical carbon dioxide fluid regulated at a temperature

10 from 31°C to 100°C and a pressure from 7.4 MPa to 30 MPa. After pouring a lubricant material into the pressure vessel, the extracting medium of supercritical carbon dioxide fluid is passed through the pressure vessel at a predetermined flow rate. The extraction process is conducted while the supercritical carbon dioxide fluid is held in a

15 state in which dissolving ability is low, that is, pressure is relatively low and temperature is relatively high, more specifically, in a state that exhibits dissolving ability for the lubricant equivalent to the ability at 60°C and 15 MPa, for example. This process extracts a component having low molecular weight and a component having end

20 groups of weak polarity, and those components are removed in this stage. Then, the extracting medium is flown at a predetermined rate. At this time, the supercritical carbon dioxide is held in a state in which dissolving ability is high, that is, the temperature is lowered while the pressure in the pressure vessel is unchanged, or the

pressure is raised while the temperature in the pressure vessel is unchanged. More specifically, the extracting fluid is held in a state that exhibits dissolving ability for the lubricant equivalent to the ability at 60°C and 20 MPa, for example. A compound with stronger polarity dissolved in the extracting fluid at that state is recovered to obtain a refined target lubricant. The residue that is remained unextracted in the vessel consists of a component with very strong polarity and a component with high molecular weight.

The refining in this method may be conducted in the temperature range from 31°C to 100°C and in the pressure range from 7.4 MPa to 30 MPa. In this range, the carbon dioxide is held in a supercritical state. The temperature range from 40°C to 100°C is preferable. At the temperatures lower than 40°C, slight change of pressure makes large variation of the dissolving ability of the supercritical carbon dioxide fluid, which makes process control difficult. On the other hand, at the temperatures higher than 100°C, only by raising the pressure much, the dissolving ability of the supercritical carbon dioxide fluid varies, which makes process control time consuming. The pressure condition is a secondary one that is determined by the decided temperature. The most preferable conditions are: the removing process is conducted under the 60°C, 15 MPa condition, and the recovering process is conducted under the 60°C, 20 MPa condition.

A lubricant used in the invention is a perfluoropolyether lubricant, and a specific example is “Fomblin Z tetraol” available from Ausimont K. K., Tokyo, Japan. “Fomblin Z tetraol” is a mixture of lubricant A having a molecular structure represented by the equation (1) and lubricant B having a molecular structure represented by the equation (2). Refining the “Fomblin Z tetraol” according to the invented method allows to remove the lubricant B with weak polarity and having only one hydroxyl group at each of the both ends and to recover the lubricant A with strong polarity and having two hydroxyl groups at each of the both ends as a target lubricant. The method provides a lubricant that contains 0 to 10 molar percent of lubricant B with respect to the mixture of the lubricants A and B.

The second aspect of the invention is a lubricant for use on a magnetic recording medium refined by the above-described method. In particular, the lubricant of the invention includes enhanced concentration of high polarity component by sorting the perfluoropolyether molecule of the lubricant based on end group structure. Ionic impurities are removed from the lubricant of the invention.

The lubricant of the invention is refined to become a composition having the end group structure with higher polarity. Thus, adhesiveness and bonding strength with the protective layer are

enhanced, leading to suppress spin migration and thermal evaporation.

Since ionic impurities, that may cause decomposition of the lubricant, are removed from the lubricant of the invention, stability of the lubricant is held during long term use by restraint of the decomposition. The ionic impurities that may be contained in the original lubricant include  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ , and  $\text{HSO}_4^-$ .

The third aspect of the invention is a magnetic recording medium using a refined lubricant described above.

A magnetic recording medium of the invention is produced by laminating a lubricant of the invention on a magnetic recording medium that is fabricated by sequentially depositing a nonmagnetic metal underlayer, a magnetic layer, and a protective layer on a substrate. Each of the underlayer, the magnetic layer, and the protective layer can be deposited by a sputtering method or a CVD method. The lubricant can be applied by a known dip-coating method.

The sputtering method, the CVD method, and the dip-coating method may be conducted using generally used equipment.

Materials for use in a magnetic recording medium of the

invention includes: an aluminum alloy substrate, a glass substrate, and a plastic substrate; a nonmagnetic underlayer of plated or sputtered NiP or NiAl; a magnetic layer containing Co, Cr, Ta, or Pt; and a nitrogen-added amorphous carbon (a-C:N) protective layer, and  
5 a diamond-like carbon (DLC) protective layer.

For enhancing adhesiveness with the protective layer, heating or UV irradiation may be conducted before or after lubricant application. An additive, for example, a cyclophosphazene compound, may be  
10 added to the lubricant.

Because the lubricant according to the present invention exhibits high adhesiveness and strong bonding with the protective layer and shows stability in long term use, a magnetic recording medium using  
15 the lubricant hardly brings about spin migration even under the condition of high rotating speed. Wear of the protective layer is minute since evaporation of lubricant by heating is little.

## Examples

20 The present invention will be described more in detail with reference to some preferred embodiments.

### Example 1

Forty gram of "Fomblin Z tetraol" (hereinafter simply referred to

as Z-tetraol) supplied from Ausimont K. K., Tokyo, Japan, is poured into a thick-walled stainless steel vessel with inner diameter of 20 mm and length of 250 mm having an inlet and an outlet, and supercritical carbon dioxide fluid was flown at a flow rate of 10 m  
 5 liter / minute. Then the temperature was held at 60°C and the pressure was varied with time. Separating at each pressure steps, the following extracted components 60-1 to 60-10 were obtained, as shown in Table 1. In this example of embodiments, the used equipment includes a liquid pump for feeding supercritical carbon  
 10 dioxide fluid, SCF-Get manufactured by JASCO Corporation, Tokyo, Japan, a full automatic pressure regulator valve, SCF-Bpg manufactured by JASCO Corporation, Tokyo, Japan, and a column oven used in a usual chromatography.

15 Table 1          Extracted weight of Z-tetraol at 60°C

| component | pressure [MPa]                    | time [min]       | weight [g] |
|-----------|-----------------------------------|------------------|------------|
| 60 - 1    | 10.0→11.0→11.5→<br>12.0→12.5→13.0 | 7→5→5→5<br>→5→20 | 3.51       |
| 60 - 2    | 14.0                              | 20               | 4.12       |
| 60 - 3    | 15.0                              | 20               | 5.38       |
| 60 - 4    | 16.0                              | 20               | 7.27       |
| 60 - 5    | 17.0                              | 20               | 6.40       |
| 60 - 6    | 18.0                              | 20               | 4.31       |
| 60 - 7    | 19.0→20.0                         | 10→10            | 3.01       |
| 60 - 8    | 22.0→24.0→26.0                    | 10→5→5           | 2.25       |
| 60 - 9    | 28.0→30.0                         | 10→20            | 2.25       |
| 60 - 10   | residue                           | ---              | 1.03       |
| total     |                                   |                  | 39.53      |

## Example 2

Forty gram of the lubricant Z-tetraol was poured into a thick-wall stainless steel vessel having an inlet and an outlet with inner diameter of 20 mm and length of 250 mm, and supercritical carbon dioxide was flown at a flow rate of 10 ml/min. Then, the temperature was held at 100°C and the pressure was varied with time.

Separating at each pressure steps, the following extracted components 100-1 to 100-11 were obtained, as shown in Table 2. In this example of embodiments, the used equipment is the same as the one used in Example 1.

Table 2      Extracted weight of Z-tetraol at 100°C

| component | pressure [MPa]                                       | time [min]                | weight [g] |
|-----------|------------------------------------------------------|---------------------------|------------|
| 100--1    | 10.0→11.0→12.0→<br>13.0→14.0→15.0→<br>16.0→17.0→18.0 | 5→5→5→5→<br>5→5→5→5→<br>5 | 0.78       |
| 100--2    | 19.0                                                 | 20                        | 2.31       |
| 100--3    | 20.0                                                 | 20                        | 3.32       |
| 100--4    | 21.0                                                 | 20                        | 4.24       |
| 100--5    | 22.0                                                 | 20                        | 6.27       |
| 100--6    | 23.0                                                 | 20                        | 7.32       |
| 100--7    | 24.0                                                 | 20                        | 6.80       |
| 100--8    | 25.0                                                 | 20                        | 3.41       |
| 100--9    | 26.0                                                 | 20                        | 1.76       |
| 100--10   | 27.0→30.0                                            | 3→20                      | 1.45       |
| 100--11   | residue                                              | ---                       | 0.71       |
| total     |                                                      |                           | 38.36      |

## 1. Testing of solubility into a solvent

Each component extracted in Example 1 and Example 2 was tested on solubility into a solvent that is generally used in a lubricant coating process, having chemical formula  $C_5F_{10}H_2$  and the trade name Vertrel XF, available from Mitsui-Dupont fluoro-chemical Co., Ltd. Each Extracted component and the solvent were mixed so that the concentration of the component is 1 weight percent. Solubility was confirmed by visual observation.

A slight amount of unsolved precipitate was observed in an unrefined lubricant material. Precipitate was also observed in components 60-9, 60-10, 100-10, and 100-11. All other extracted components were dissolved including from 60-1 to 60-8 and from 100-1 to 100-9.

## 2. NMR measurement

Molar compositions of lubricant A and lubricant B were measured for the components 60-1 to 60-10 by means of  $F_{19}$ -NMR. The results are shown in Table 3.

Table 3      Mixing ratio of lubricants for the components extracted at 60° C.

| component | lubricant A (%) | lubricant B (%) |
|-----------|-----------------|-----------------|
| 60--1     | 63              | 37              |
| 60--2     | 70              | 30              |
| 60--3     | 82              | 18              |
| 60--4     | 87              | 13              |
| 60--5     | 94              | 6               |
| 60--6     | 95              | 5               |
| 60--7     | 100             | 0               |
| 60--8     | 100             | 0               |
| 60--9     | 100             | 0               |
| 60--10    | 100             | 0               |

The structure of the lubricant B was more contained in the components extracted at lower pressures. Content of lubricant A can be increased by removing the components extracted at lower pressure. Dissolving ability of the supercritical carbon dioxide fluid for a polar component is known to decrease in conditions of temperature pressure at which the density of the supercritical carbon dioxide fluid is low. The lubricant B exhibits weaker polarity because it has fewer hydroxyl groups per molecule as compared with the lubricant A. Therefore, the lubricant B was predominantly dissolved into the supercritical carbon dioxide fluid at relatively low pressure.

Under the same pressure condition, the density of the supercritical carbon dioxide fluid decreases at higher temperatures.

When extraction process is conducted at 100° C in place of 60° C, the dissolving ability of the fluid for a polar component is lower at the same pressure. Therefore, it is understood that higher pressure is generally required to dissolve the same amount of lubricant at 100° C than at 60° C

### 3. Measurement of ionic impurities

Several microgram of unrefined material and each of the extracted components 60-1 to 60-10 and 100-1 to 100-11 were dropped on a surface of a magnetic recording medium uncoated with lubricant. Quantity of each kind of ions on the surface was measured by means of TOF-SIMS. The results are given in Table 4. The numerical values in the Table are the detected quantity that is number of counts per unit time in the TOF-SIMS.

The results show that cations are frequently detected in the component extracted at higher pressures and the residue after extraction.  $\text{HCO}_3$  and  $\text{HSO}_4$  are frequently detected in the residue. Chromiums are detected in the component extracted at high pressure side and also in the low pressure extraction components 60-1, 2, 3 and 100-1, 2, 3, 4. In the 60-1, 2, 3 and 100-1, 2, 3, 4,  $\text{CF}_2\text{Cl}$  was at the same time detected. But, the  $\text{CF}_2\text{Cl}$  was not detected in the high pressure extraction components and the residue. Ionic impurities, which are components with fairly strong polarity, either slightly

dissolves in a relatively low temperature and high pressure state, or do not dissolve in the fluid at a pressure of up to 30 MPa leaving in a residue. Consequently, it can be understood that those components may be removed together with the residue in a refinement using  
5 supercritical carbon dioxide fluid as an extraction medium.

Chromium was detected in low pressure components, as well. But, they are supposed not to present in the form of ions. The reason is that  $\text{CF}_2\text{Cl}$  was detected by close observation of fragments of the  
10 TOF-SIMS, which may indicate that the extracted molecules are impurities having end group of  $-\text{CF}_2\text{Cl}$  in place of  $-\text{CH}_2\text{OH}$  in the lubricant B.

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Table 4      Number of counts for each type of ions detected by means of TOF-SIMS

| component | cation |      | anion |                  |                  |
|-----------|--------|------|-------|------------------|------------------|
|           | K      | Na   | Cl    | HCO <sub>3</sub> | HSO <sub>4</sub> |
| unrefined | 45684  | 925  | 1793  | 57               | 462              |
| 60--1     | 304    | 56   | 3110  | 11               | 20               |
| 60--2     | 220    | 42   | 2040  | 3                | 39               |
| 60--3     | 171    | 20   | 1381  | 5                | 19               |
| 60--4     | 136    | 14   | 289   | 3                | 24               |
| 60--5     | 214    | 24   | 144   | 14               | 36               |
| 60--6     | 425    | 22   | 259   | 4                | 34               |
| 60--7     | 1211   | 51   | 193   | 9                | 21               |
| 60--8     | 6154   | 190  | 264   | 7                | 42               |
| 60--9     | 7157   | 333  | 519   | 15               | 74               |
| 60--10    | 107611 | 3269 | 2236  | 115              | 353              |
| 100--1    | 7261   | 215  | 5399  | 15               | 79               |
| 100--2    | 736    | 40   | 3544  | 5                | 18               |
| 100--3    | 350    | 16   | 2624  | 4                | 17               |
| 100--4    | 433    | 32   | 2154  | 7                | 25               |
| 100--5    | 552    | 20   | 937   | 2                | 23               |
| 100--6    | 531    | 15   | 340   | 4                | 27               |
| 100--7    | 349    | 7    | 263   | 0                | 25               |
| 100--8    | 837    | 19   | 261   | 5                | 25               |
| 100--9    | 1563   | 28   | 204   | 6                | 21               |
| 100--10   | 4574   | 69   | 455   | 12               | 40               |
| 100--11   | 102855 | 4584 | 2060  | 161              | 452              |

#### 4. Bonded ratio in a lubricant film and loss by heating

5      A nonmagnetic substrate of aluminum - magnesium alloy was prepared. After forming a nonmagnetic layer by plating Ni-P by means of electroless plating, the surface was polished. Nearly concentric circular grooves were made having a surface roughness Ra

of 0.8 nm by means of texturing using diamond slurry. After cleaning the substrate, a nonmagnetic metal underlayer of chromium was formed in a commonly used sputtering apparatus. Subsequently, a magnetic layer containing Co, Cr, Ta, and Pt and a protective layer of nitrogen-added amorphous carbon (a-C:N) having thickness of 8 nm were formed by a CVD method. This medium was employed as a common medium. Each of the common media was coated with unrefined Z-tetraol or each of the extracted components 60-1 to 60-9. The coating was conducted by dipping method using a solution including 0.06 wt% of the extracted lubricant in the solvent of Vertrel XF. Speed of pulling-up was controlled between 0.5 and 3 mm/s so as to adjust the thickness of the lubricant film to 2.4 nm. Thickness of the lubricant film was measured on the produced samples by means of FT-IR method. Then, mobile component of the lubricant film was removed using fluorine-containing solvent. The thickness of the left component of the lubricant film was measured as bonded component of the lubricant film. Bonded ratio of the lubricant film is defined by the thickness of the bonded component of the lubricant film divided by the total thickness of the lubricant film.

Each magnetic recording medium was heated at 140°C for 60 minutes, and the film thickness was measured after the heating to obtain a loss by heating. Those values are given in Table 5.

The components that include relatively large quantity of the lubricant B exhibited low bonded ratio, and the bonded ratio increased as the quantity of the lubricant B decreased. The loss by heating showed similar tendency.

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Table 5      Bonded ratio on a magnetic recording medium and loss by heating

| component | bonded ratio (%) | loss by heating (nm) |
|-----------|------------------|----------------------|
| unrefined | 40.4             | -0.51                |
| 60--1     | 31.5             | -0.74                |
| 60--2     | 33.3             | -0.54                |
| 60--3     | 32.5             | -0.30                |
| 60--4     | 37.0             | -0.28                |
| 60--5     | 42.6             | -0.24                |
| 60--6     | 46.9             | -0.26                |
| 60--7     | 50.6             | -0.23                |
| 60--8     | 54.1             | -0.10                |
| 60--9     | 53.5             | -0.04                |

### Example 3

10      Forty gram of the lubricant Z-tetraol was poured into a thick-wall stainless steel vessel having an inlet and an outlet with inner diameter of 20 mm and length of 250 mm, and supercritical carbon dioxide fluid was flown at a flow rate of 10 mL/min. The component dissolved in the fluid at 60°C and 15 MPa during 60 minutes was  
15 removed. From the left residue, the component dissolved in the fluid at 60°C and 20 MPa flown at the flow rate of 10 mL/min for 40

minutes was recovered to obtain 20 g of a material for lubricant.

In this Example, the same equipment as the one used in Example 1 was used.

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Molar composition of the lubricant A and B in the obtained lubricant was measured by means of NMR and resulted in A: 91% and B: 9%. A magnetic recording medium having a lubricant film 2.4 nm thick was produced using this lubricant. Bonded ratio and loss by heating were measured resulting in bonded ratio of 45% and the loss by heating of 0.25 nm. Quantity of each kind of ions present on the completed magnetic recording medium was measured by means of TOF-SIMS. All of Na, K, Cl,  $\text{HCO}_3$ , and  $\text{HSO}_4$  decreased to less than 10% with respect to those of a magnetic recording medium using unrefined lubricant.

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#### Advantageous Effect of the invention

As described so far, by employing a lubricant in which a bonded ratio is enhanced, a loss by heating is suppressed, and ionic impurities are decreased, a magnetic recording medium can be provided that exhibits excellent lubricating performance and stability during long term use under a circumstance of high rotating speed and accompanied high temperature of the magnetic recording medium mounted on a fixed magnetic recording device.

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What is claimed is:

1. A method to refine a lubricant including a compound A having a perfluoropolyether molecular structure that has two hydroxyl groups at each  
5 of two ends of compound A, and a compound B having the perfluoropolyether molecular structure that has one hydroxyl group at each of two ends of compound B, the method comprising:

placing the lubricant into a pressure vessel set at a predetermined pressure and at a predetermined temperature;

10 removing the compound B by a process comprising: flowing an extracting medium of a supercritical carbon dioxide fluid at a predetermined rate through the pressure vessel,

holding the pressure vessel at the predetermined temperature and the predetermined pressure,

15 dissolving the compound B in the supercritical carbon dioxide fluid, and

removing the dissolved compound B from the pressure vessel; and recovering the compound A by a process comprising:

20 lowering the extracting medium of the supercritical carbon dioxide fluid through the pressure vessel,

holding the pressure vessel at the predetermined pressure and at a temperature lower than the predetermined temperature,

dissolving the compound A in the extracting medium, and recovering the dissolved compound A from the pressure vessel.

25 2. The method to refine a lubricant as recited in claim 1, wherein the

predetermined temperature is in a range from 31°C to 100°C and the predetermined pressure is in a range from 7.4 MPa to 30 MPa.

3. The method to refine a lubricant as recited in claim 2, wherein the predetermined temperature is in a range from 40°C to 100°C.

4. The method to refine a lubricant as recited in claim 1, wherein the compound A is a lubricant A represented by formula (1) and the compound B is a lubricant B represented by formula (2);

HO-CH<sub>2</sub>-CH(OH)-CH<sub>2</sub>-O-CH<sub>2</sub>-CF<sub>2</sub>-(O-C<sub>2</sub>F<sub>4</sub>)<sub>p</sub>-(O-CF<sub>2</sub>)<sub>q</sub>-O-CF<sub>2</sub>-CH<sub>2</sub>-O-CH<sub>2</sub>-CH(OH)-CH<sub>2</sub>-OH

lubricant A, formula (1):

HO-CH<sub>2</sub>-CF<sub>2</sub>-(O-C<sub>2</sub>F<sub>4</sub>)<sub>p</sub>-(O-CF<sub>2</sub>)<sub>q</sub>-O-CF<sub>2</sub>-CH<sub>2</sub>-OH,

where p and q are positive integers.

lubricant B, formula (2):

5. A method to refine a lubricant including a compound A having a perfluoropolyether molecular structure that has two hydroxyl groups at each of two ends of compound A, and a compound B having the perfluoropolyether molecular structure that has one hydroxyl group at each of two ends of compound B, the method comprising:

placing the lubricant into a pressure vessel set at a predetermined pressure and at a predetermined temperature;

removing the compound B by a process comprising:

flowing an extracting medium of a supercritical carbon dioxide fluid at a predetermined rate through the pressure vessel,

holding the pressure vessel at the predetermined temperature and at the predetermined pressure,

dissolving the compound B in the supercritical carbon dioxide fluid, and

5 removing the dissolved compound B from the pressure vessel; and recovering the compound A by a process comprising:

flowing the extracting medium of the supercritical carbon dioxide fluid through the pressure vessel,

holding the pressure vessel at the predetermined temperature and at a pressure higher than the predetermined pressure,

10 dissolving the compound A in the supercritical carbon dioxide fluid, and

recovering the dissolved compound A from the pressure vessel.

15 6. The method to refine a lubricant as recited in claim 5, wherein the predetermined temperature is in a range from 31°C to 100°C and the predetermined pressure is in a range from 7.4 MPa to 30 MPa.

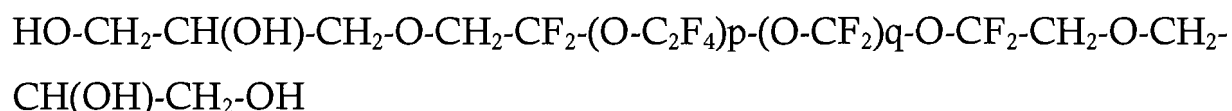
7. The method to refine a lubricant as recited in claim 6, wherein the predetermined temperature is in a range from 40°C to 100°C.

8. The method to refine a lubricant as recited in claim 5, wherein the removing of the compound B comprises removing a component that is dissolved in the supercritical carbon dioxide fluid under a state at 60°C and 15 Mpa.

9. The method to refine a lubricant as recited in claim 5, wherein the recovering of the compound A comprises recovering a component that is dissolved in the supercritical carbon dioxide fluid under a state at 60° C and 20 MPa.

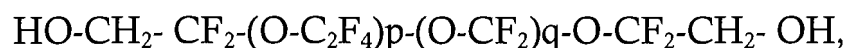
5

10. The method to refine a lubricant as recited in claim 5, wherein the compound A is a lubricant A represented by formula (1) and the compound B is a lubricant B represented by formula (2),



10

lubricant A, formula (1):



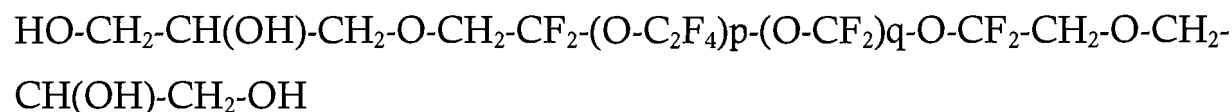
lubricant B, formula (2):

where p and q are positive integers.

15

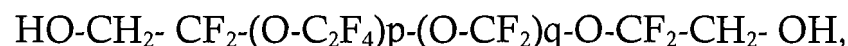
11. The method to refine a lubricant as recited in claim 5, wherein the lubricant is a mixture of a lubricant A having a molecular structure represented by a formula (1) and a lubricant B having a molecular structure represented by a formula (2), the mixture comprising the lubricant B in an amount from zero to 10 molar percent with respect a molar amount of the mixture of the lubricants A and B,

20



lubricant A, formula (1):

25



lubricant A, formula (2):

where p and q are positive integers.

12. A lubricant of a magnetic recording medium, comprising:

a compound A comprising a perfluoropolyether molecular structure  
5 having two hydroxyl groups at each of two ends of compound A; and

a compound B comprising the perfluoropolyether molecular structure  
having one hydroxyl group at each of two ends of compound B, wherein the  
lubricant is refined by

placing the lubricant into a pressure vessel set at a predetermined  
10 pressure and a predetermined temperature,

removing the compound B by a process comprising:

flowing an extracting medium of a supercritical carbon dioxide fluid at  
a predetermined rate through the pressure vessel,

holding the pressure vessel at the predetermined temperature and at  
15 the predetermined pressure,

dissolving the compound B in the supercritical carbon dioxide fluid,  
and

removing the dissolved compound B from the pressure vessel, and  
recovering the compound A by a process comprising:

20 flowing the extracting medium of the supercritical carbon dioxide fluid  
through the pressure vessel,

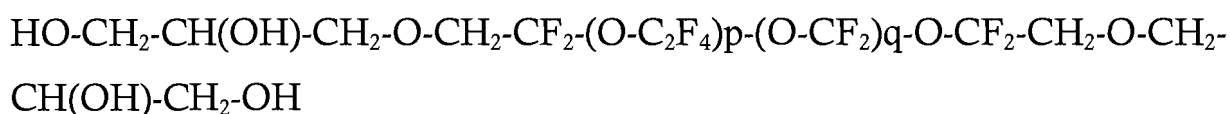
holding the pressure vessel at the predetermined pressure and at a  
temperature lower than the predetermined temperature,

dissolving the compound A in the supercritical carbon dioxide fluid,

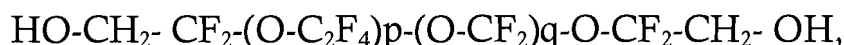
25 and

recovering the dissolved compound A from the pressure vessel.

13. The lubricant of the magnetic recording medium as recited in claim 12, wherein the lubricant is a mixture of a lubricant A having a molecular structure represented by a formula (1) and a lubricant B having a molecular structure represented by a formula (2), the mixture comprising the lubricant B in an amount from zero to 10 molar percent with respect a molar amount of the mixture of the lubricants A and B:



lubricant A, formula (1):



lubricant B, formula (2):

where p and q are positive integers.

14. A magnetic recording medium using a lubricant, the lubricant comprising:

a compound A comprising a perfluoropolyether molecular structure having two hydroxyl groups at each of two ends of compound A; and

a compound B comprising the perfluoropolyether molecular structure having one hydroxyl group at each of two ends of compound B, wherein the

lubricant is refined by

placing the lubricant into a pressure vessel set at a predetermined pressure and at a predetermined temperature,

removing the compound B by a process comprising:

flowing an extracting medium of a supercritical carbon dioxide fluid at

a predetermined rate through the pressure vessel,

holding the pressure vessel at the predetermined temperature and at

the predetermined pressure,

dissolving the compound B in the supercritical carbon dioxide fluid,  
and

removing the dissolved compound B from the pressure vessel, and  
5 recovering the compound A by a process comprising:

flowing the extracting medium of the supercritical carbon dioxide fluid  
through the pressure vessel,

holding the pressure vessel at the predetermined pressure and at a  
temperature lower than the predetermined temperature,

10 dissolving the compound A in the supercritical carbon dioxide fluid,  
and

recovering the dissolved compound A from the pressure vessel.

15 15. The magnetic recording medium as recited in claim 14, wherein the  
lubricant is a mixture of a lubricant A having a molecular structure  
represented by a formula (1) and a lubricant B having a molecular structure  
represented by a formula (2), the mixture comprising the lubricant B in an  
amount from zero to 10 molar percent with respect a molar amount of the  
mixture of the lubricants A and B:

20  $\text{HO-CH}_2\text{-CH(OH)-CH}_2\text{-O-CH}_2\text{-CF}_2\text{-(O-C}_2\text{F}_4\text{)}_p\text{-(O-CF}_2\text{)}_q\text{-O-CF}_2\text{-CH}_2\text{-O-CH}_2\text{-}$   
 $\text{CH(OH)-CH}_2\text{-OH}$

lubricant A, formula (1):

$\text{HO-CH}_2\text{-CF}_2\text{-(O-C}_2\text{F}_4\text{)}_p\text{-(O-CF}_2\text{)}_q\text{-O-CF}_2\text{-CH}_2\text{-OH,}$

lubricant B, formula (2):

25 where p and q are positive integers.

16. A lubricant of a magnetic recording medium, comprising:

a compound A comprising a perfluoropolyether molecular structure having two hydroxyl groups at each of two ends of compound A; and

a compound B comprising the perfluoropolyether molecular structure having one hydroxyl group at each of two ends of compound B, wherein the lubricant is refined by

placing the lubricant into a pressure vessel set at a predetermined pressure and at a predetermined temperature,

removing the compound B by a process comprising:

flowing an extracting medium of a supercritical carbon dioxide fluid at a predetermined rate through the pressure vessel,

holding the pressure vessel at the predetermined temperature and at the predetermined pressure,

dissolving the compound B in the supercritical carbon dioxide fluid,

and

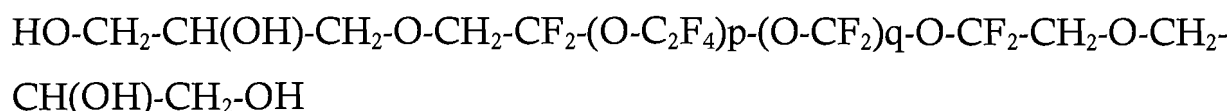
removing the dissolved compound B from the pressure vessel, and recovering the compound A by a process comprising:

flowing the extracting medium of the supercritical carbon dioxide fluid through the pressure vessel, holding the pressure vessel at the predetermined temperature and at a pressure higher than the predetermined pressure

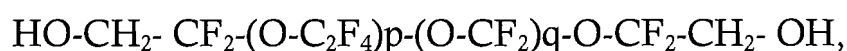
dissolving the compound A in the extracting medium, and recovering the dissolved compound A from the pressure vessel.

17. The lubricant of the magnetic recording medium as recited in claim 16, wherein the lubricant is a mixture of a lubricant A having a molecular structure represented by a formula (1) and a lubricant B having a molecular

structure represented by a formula (2), the mixture comprising the lubricant B in an amount from zero to 10 molar percent with respect a molar amount of the mixture of the lubricants A and B:



lubricant A, formula (1):



lubricant B, formula (2):

where p and q are positive integers.

18. The lubricant of the magnetic recording medium as recited in claim 16, wherein the predetermined temperature is in a range from 31°C to 100°C and the predetermined pressure is in a range from 7.4 MPa to 30 MPa.

19. The lubricant of the magnetic recording medium as recited in claim 18, wherein the predetermined temperature is in a range from 40°C to 100°C.

20. A magnetic recording medium using a lubricant, the lubricant comprising:

a compound A comprising a perfluoropolyether molecular structure having two hydroxyl groups at each of two ends of compound A; and

a compound B comprising the perfluoropolyether molecular structure having one hydroxyl group at each of two ends of compound B, wherein the lubricant is refined by

placing the lubricant into a pressure vessel set at a predetermined pressure and at a predetermined temperature,

removing the compound B by a process comprising:

flowing an extracting medium of a supercritical carbon dioxide fluid at a predetermined rate through the pressure vessel,

holding the pressure vessel at the predetermined temperature and at the predetermined pressure,

dissolving the compound B in the supercritical carbon dioxide fluid, and

removing the dissolved compound B from the pressure vessel, and

recovering the compound A by a process comprising:

flowing the extracting medium of the supercritical carbon dioxide fluid through the pressure vessel,

holding the pressure vessel held at the predetermined temperature and at a pressure higher than the predetermined pressure,

dissolving the compound A in the supercritical carbon dioxide fluid,

and

recovering the dissolved compound A from the pressure vessel.

21. The magnetic recording medium as recited in claim 20, wherein the lubricant is a mixture of a lubricant A having a molecular structure represented by a formula (1) and a lubricant B having a molecular structure represented by a formula (2), the mixture comprising the lubricant B in an amount from zero to 10 molar percent with respect a molar amount of the mixture of the lubricants A and B:

$$\text{HO-CH}_2\text{-CH(OH)-CH}_2\text{-O-CH}_2\text{-CF}_2\text{-(O-C}_2\text{F}_4\text{)}_p\text{-(O-CF}_2\text{)}_q\text{-O-CF}_2\text{-CH}_2\text{-O-CH}_2\text{-CH(OH)-CH}_2\text{-OH}$$

lubricant A, formula (1):

HO-CH<sub>2</sub>-CF<sub>2</sub>-(O-C<sub>2</sub>F<sub>4</sub>)<sub>p</sub>-(O-CF<sub>2</sub>)<sub>q</sub>-O-CF<sub>2</sub>-CH<sub>2</sub>-OH,

lubricant B, formula (2):

where p and q are positive integers.

- 5    22. The magnetic recording medium as recited in claim 20, wherein the predetermined temperature is in a range from 31°C to 100°C and the predetermined pressure is in a range from 7.4 MPa to 30 MPa.

23. The magnetic recording medium as recited in claim 22, wherein the  
10    predetermined temperature is in a range from 40°C to 100°C.