



Method for refining lubricant for a magnetic recording medium

Technical field of the invention

The present invention relates to a method for refining lubricant
 5 for a magnetic recording medium, lubricant refined by the method,
 and a magnetic recording medium using the lubricant.

Background of the invention

Recording media such as fixed magnetic recording media are
 10 used as general-purpose data recording media in computers. A
 magnetic recording apparatus containing fixed magnetic recording
 media comprises a driving mechanism for the magnetic recording
 media, a driving mechanism for a magnetic head, a stopping
 mechanism for the magnetic head, and a data transfer mechanism as
 15 well as one or more magnetic recording media. In recent years, the
 magnetic recording media are showing significant progress in
 increasing recording density, capacity, and data transfer rate.

Conventional fixed magnetic recording apparatus employs the
 20 contact start and stop (CSS) system, in which a magnetic recording
 head floats while the magnetic recording medium is rotating and the
 magnetic head becomes in contact with the surface of the magnetic
 recording medium when the motor for driving rotation of the
 magnetic recording medium stops. In this system, the magnetic head



slides on the surface of the magnetic recording medium at the time of start and stop of rotation of the magnetic recording medium, and friction occurs between the magnetic recording medium and the magnetic head. In order to protect the magnetic layer from the friction, a protective layer is provided, and a lubricant layer is provided to improve lubricity on the surface.

Recently, rotating speed of a magnetic recording medium of a magnetic recording apparatus has increased from traditional 5,400 rpm to a speed in a range from 7,200 to 15,000 rpm. As a result, a spin migration or spin off phenomenon has become significant, in which lubricant on the surface of a magnetic recording medium moves to a peripheral region or scatters from the medium surface. Furthermore, the heat generated at high rotating speed evaporates the lubricant and raises a problem of loss of the lubricant mass.

The thickness growth of the lubricant layer in the peripheral region of the magnetic recording medium induced by the spin migration causes adhesion obstruction. The evaporation of the lubricant is liable to cause wear of the protective layer because of the decrease of the lubricant layer thickness. In the worst case, head crash occurs.

A lubricant layer of a magnetic recording medium needs to be

stably formed in a uniform thickness on a protective layer surface to avoid spin migration. In addition, strong adhesion and bonding are required at the interface between the lubricant layer and the protective layer. To enhance the adhesiveness, perfluoropolyether lubricant having a terminal group of a hydroxyl group or a piperonyl group is used. These types of lubricant are available, for example, from Ausimont KK under the trade names of Fomblin Z DOL, Fomblin Z tetraol, and AM3001. Most of the presently used perfluoropolyether lubricant is prone to exhibit poor heat resistance in a very low molecular weight range and high statical friction in a very high molecular weight range. Consequently, lubricant now used has a number average molecular weight (M_n) in the range from 1,000 to 10,000, more specifically, from 2,000 to 4,000.

The above-described kinds of lubricant perform well in the range of present rotating speed of a magnetic recording medium. However, at higher rotating speed, there remains fear about durability against a phenomenon like spin migration.

Japanese Unexamined Patent Application Publication No. 2001-229524 discloses use of lubricant that has high molecular weight and narrow distribution of molecular weight. Such lubricant eliminated the problem accompanied by conventional lubricant having high molecular weight and increased adhesiveness with a protective layer,

improving durability against spin migration.

While a lubricant having high molecular weight was used in the Publication, minimized flying height of a magnetic head in these days
5 raises a problem by an effect of the lubricant molecules. Namely, the size of the lubricant molecules has some effect on the flight of the magnetic head in a circumstance of a minimized distance between the magnetic head and the magnetic recording medium. Accordingly, if the durability against the spin migration is similar, lubricant with
10 lower molecular weight is preferable as long as heat resistance is secured.

As described above, lubricant needs improvement in view of molecular weight, durability against spin migration, and heat
15 resistance.

Besides the above problems, commercially available lubricant contains impurities, which needs to be eliminated. For example, commercially available perfluoropolyether lubricant contains
20 impurities of perfluoropolyether having a terminal group such as CF_2Cl , CF_3 , or CF_2H . These terminal groups exhibit weaker polarity as compared with a hydroxyl group and a piperonyl group, which are the terminal groups intrinsic to perfluoropolyether lubricant. If the content of those impurities is large, adhesion and binding with a

protective layer cannot be improved even if molecular weight is large. Therefore, a refinement method to eliminate this kind of impurities is required.

- 5 Japanese Unexamined Patent Application Publication No. H5-20673 discloses a method for refining lubricant, in which a technique is performed for refinement to control molecular weight by means of a gel permeation chromatography (GPC) method. Japanese Unexamined Patent Application Publication No. H11-199882
- 10 discloses a technique for refinement by controlling molecular weight of lubricant using an organic solvent. A method for refining lubricant using an organic solvent tends to predominantly eliminate highly polar and low molecular weight components. Consequently, the lubricant refined by the method possibly exhibits increased
- 15 proportion of the perfluoropolyether having terminal group of CF_2Cl , CF_3 , or CF_2H that shows low solubility in an organic solvent. Japanese Unexamined Patent Application Publication No. 2001-164279 discloses a refinement method to increase proportion of molecules having the terminal groups that are intrinsic to
- 20 perfluoropolyether lubricant by applying supercritical fluid chromatography (SFC). While the reference states that the method is less expensive than a method using an organic solvent and performs higher productivity than a GPC method, problems may arise in the cost and durability of a packing column in the SFC method when the

mass treated in one process becomes large.

Further, commercially available lubricant also includes ionic impurities as well as the above-described perfluoropolyether of weak polarity. The ionic impurities included in lubricant existing on a recording medium causes corrosion, which induces dissolution of ions of metallic element such as cobalt used in a magnetic layer. The cobalt ions catalyze decomposition of perfluoropolyether molecules used in the lubricant. Therefore, the ionic impurities are desired to be eliminated to the possible minimum quantity.

Thus, the ionic impurities that have entered into perfluoropolyether in its synthesis process are required to be eliminated.

Problem to be solved by the invention

An object of the present invention is to provide a method for refining lubricant that exhibits excellent lubrication performance and high stability in long term use, and applicable to a magnetic recording medium. Another object of the present invention is to provide lubricant that exhibits excellent lubrication performance and high stability in long term use by means of such method for refinement. Still another object of the present invention is to provide a magnetic recording medium that exhibits excellent lubrication

performance and high stability in long term use applying such lubricant.

Means to solve the problem

5 The above objects are achieved by the present invention as described below.

10 The first invention is a method for refining perfluoropolyether lubricant by means of supercritical extraction method using carbon dioxide in a supercritical fluid state (called as “supercritical carbon dioxide” in this specification).

15 The first mode of this first invention is a method for refining lubricant containing a compound having perfluoropolyether structure by means of supercritical extraction, wherein the lubricant is contacted with extracting medium of supercritical carbon dioxide under a predetermined condition in a pressure vessel to remove ionic impurities from the lubricant. This mode of the refinement method is conducted under a predetermined condition at such temperature and 20 pressure that the density of the supercritical carbon dioxide is not larger than the density at the temperature of 60°C and the pressure of 20 MPa. The density in the boundary state is about 0.7 g/cm³. The ionic impurities in this mode of refinement method can be sodium ions, potassium ions, chloride ions, HCO₃ ions, HSO₄ ions, or

sulfuric ions.

The second mode of this first invention is a method for refining lubricant containing a compound having perfluoropolyether structure
5 by means of supercritical extraction, wherein the lubricant is contacted with extracting medium of supercritical carbon dioxide under a predetermined condition in a pressure vessel to remove perfluoropolyether compounds having a terminal group of weak polarity out of the components of the perfluoropolyether lubricant.
10 This mode of the refinement method is conducted under a predetermined condition at such temperature and pressure that the density of the supercritical carbon dioxide is not larger than the density at the temperature of 60°C and the pressure of 16 MPa. The density in the boundary state is about 0.6 g/cm³. The
15 perfluoropolyether compounds having a terminal group of weak polarity in this second mode of refinement method of the invention have a terminal group of weak polarity such as CF₃, CF₂H, or CF₃Cl.

Third mode of this first invention is a method for refining
20 lubricant containing a compound having perfluoropolyether structure by means of supercritical extraction, wherein the lubricant is contacted with extracting medium of supercritical carbon dioxide under a first condition in a pressure vessel to remove perfluoropolyether compounds having a terminal group of weak

polarity out of the components of the perfluoropolyether lubricant, and then, the lubricant from which perfluoropolyether compounds having a terminal group of weak polarity have been removed is contacted with extracting medium of supercritical carbon dioxide under a second condition in the pressure vessel to remove ionic impurities and to extract and recover refined perfluoropolyether lubricant. In this mode of this invention, the first condition is a combination of a temperature and a pressure at which a density of the supercritical carbon dioxide is not larger than the density of temperature of 60°C and the pressure of 16 MPa (the density is about 0.6 g/cm³) and the second condition is a combination of a temperature and a pressure at which a density of the supercritical carbon dioxide is not larger than the density at the temperature of 60°C and the pressure of 20 MPa (the density is about 0.7 g/cm³). In this mode of the invention, the perfluoropolyether compound having a terminal group of weak polarity has a terminal group of weak polarity such as CF₃, CF₂H, or CF₂Cl, and the ionic impurities can be sodium ions, potassium ions, chloride ions, HCO₃ ions, HSO₄ ions, or sulfuric ions.

The second invention is a perfluoropolyether lubricant that is refined by the method for refining lubricant of above-described first invention.

The third invention is a magnetic recording medium comprising

nonmagnetic underlayer, magnetic layer, protective layer, and a lubricant layer that are sequentially laminated on a magnetic substrate, wherein the lubricant layer is composed of the above-described perfluoropolyether lubricant of the second invention.

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Brief description of drawings

Fig. 1 is a schematic cross sectional view of a magnetic recording medium according to the present invention.

Fig. 2 is a graph showing SFC extraction of the lubricant AM65-2.

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Aspects of embodiments of the present invention

The present invention will be described below. The first invention is described first. The first invention is a method for refining perfluoropolyether lubricant by means of supercritical extraction using supercritical carbon dioxide.

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Supercritical fluid continuously varies from a quite dilute low density state to a liquid-like high density state with slight change of temperature and pressure. Since dissolving ability of supercritical fluid depends on this density, the dissolving ability can be freely varied by controlling temperature and pressure.

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The refinement method of the invention can be conducted in the temperature range from 31°C to 100°C. While the pressure of 7.4

MPa is sufficient, the method can be performed in the pressure range up to 30 MPa for the above-mentioned temperature range. Within this range, carbon dioxide is held in a supercritical state.

Particularly favorable temperature range in the invention is from 40°C to 100°C. At a temperature below 40°C, small variation of pressure greatly changes dissolving ability of the supercritical carbon dioxide, resulting in difficulty in control. At a temperature over 100°C, large variation of pressure is necessary for varying dissolving ability of the supercritical carbon dioxide, taking much time for control. The above-specified temperature and pressure range corresponds to the range of about 0.4 g/cm³ to about 0.9 g/cm³ of the density of the supercritical carbon dioxide. In the present invention, the dissolving ability can be freely changed in the range corresponding to the above-described temperature and pressure range by controlling these parameters. Consequently, a state of the supercritical carbon dioxide having a desired density, which corresponds to desired dissolving ability, can be generated by controlling temperature and pressure. Perfluoropolyether lubricant is contacted with this supercritical carbon dioxide to extract a desired lubricant component, in the present invention. Refinement of lubricant in the present invention can be performed based on differences in molecular weight or functional groups of the perfluoropolyether, or small difference in the properties, e.g., polarity, of compounds or ions contained in the lubricant.

Refining methods of the present invention can be classified into first, second, and third modes according to the component to be removed and the component to be extracted, as described below.

5 The first mode of this invention will be described first. The first mode of this invention is a method for refining perfluoropolyether lubricant removing ionic impurities from the lubricant.

10 Ionic impurities, when contained in the lubricant, decompose the lubricant, raising a problem in long term stability.

Accordingly, long term stability of the lubricant is secured by removing ionic impurities and preventing the lubricant from decomposing. The removal of ionic impurities from lubricant is performed by extraction using supercritical carbon dioxide at a
15 density not larger than a predetermined upper limit.

20 Ionic impurities that can be removed by a method of the invention include metallic ions, for example sodium ions and potassium ions, and inorganic ions, for example, chloride ions, HCO_3 ions, HSO_4 ions, and sulfuric ions. In addition, organic ions, for example, ammonium ions, oxalic ions, and formic ions, can be removed as well in the present invention.

Specific description of the first mode is given below.

The first mode is a method for refining perfluoropolyether lubricant by means of supercritical extraction, wherein the lubricant is contacted with extracting medium of supercritical carbon dioxide under a predetermined condition in a pressure vessel to remove ionic
5 impurities from the lubricant. More specifically, this mode of refinement method comprises the following steps.

- (a) A step of introducing perfluoropolyether lubricant into a pressure vessel,
- 10 (b) a step of contacting the lubricant with supercritical carbon dioxide under a predetermined condition in the pressure vessel to extract a perfluoropolyether compounds, and
- (c) a step of recovering the perfluoropolyether compounds obtained in the step (b).

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This mode is described below following these steps.

The method of the present invention is a supercritical extraction method using an extraction medium of supercritical carbon dioxide. This method is based on the fact that the solubility of a polar
20 component in supercritical carbon dioxide can be suppressed to a low level in a certain condition.

Step (a)

Raw material of perfluoropolyether lubricant is introduced into a

pressure vessel.

The pressure vessel comprises an inlet part through which an extracting medium of supercritical carbon dioxide is introduced, an outlet part through which the medium and extracted substance are taken out, and a control part that controls the temperature and pressure in the vessel at a desired value. A vessel and a device that meet the above requirements include, for example, a stainless steel vessel with thick wall having inlet and outlet ports, a pump for circulating supercritical carbon dioxide, for example, SCF-Get manufactured by JASCO Corporation, a full automatic pressure regulator valve, for example, SCF-Bpg manufactured by JASCO Corporation, and a column oven used in common chromatogram. Capacity of the pressure vessel may be selected corresponding to the lubricant to be refined, and is not limited to any special value.

Lubricant that can be treated with in a refinement method of the present invention is perfluoropolyether lubricant having a polar group, for example, a piperonyl group, a hydroxyl group, a carboxyl group, an ester group, an amino group, or a cyclophosphazene group. Specific examples of such lubricant are 'Fomblin Z tetraol', and 'AM3001' commercially available from Ausimont KK. A predetermined quantity of such a raw material is introduced into a pressure vessel through an inlet part of the vessel.

Step (b)

The lubricant introduced in the pressure vessel is contacted with an extracting medium of supercritical carbon dioxide and perfluoropolyether compounds are extracted removing ionic
5 impurities.

The extracting medium is supercritical carbon dioxide. The supercritical carbon dioxide flows through the pressure vessel at a predetermined rate and contacts with the raw lubricant in the vessel.
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While the step (b) can be conducted in the temperature and pressure range corresponding to the density range of 0.4 to 0.9 g/cm³ as mentioned previously, the step is preferably conducted within more confined temperature and pressure in that range. The
15 preferable temperature and pressure in conformity with this mode of the invention are the temperature and pressure that allow the state with the density of the supercritical carbon dioxide of not larger than about 0.7 g/cm³. Specific examples of the boundary temperature and pressure can be 60°C, 20 MPa and 100°C, 26 MPa. By contacting the
20 lubricant with the supercritical carbon dioxide under such a condition, perfluoropolyether compound components are selectively extracted remaining ionic impurities in the residue in the vessel.

A flow rate of the supercritical carbon dioxide in this step varies

with the extraction condition and a target compound to be extracted, and is not limited to a special value. However, a flow rate that allows enough contact between the lubricant and the supercritical carbon dioxide is desired. A preferable flow rate is in the range from 1
5 mL/min to 100 mL/min per 100 g of the lubricant, more preferably, from 10 mL/min to 50 mL/min per 100 g of the lubricant. Excessively low flow rate causes a problem of low processing speed. Excessively high flow rate causes decrease of extraction efficiency and raises a problem of unnecessary consumption and a problem that the
10 lubricant is driven out unless the vessel volume is increased.

Step (c).

The extracted perfluoropolyether compounds are collected together with the supercritical carbon dioxide to recover the
15 perfluoropolyether compounds. The perfluoropolyether compounds and the supercritical carbon dioxide are taken out through an outlet port of the pressure vessel. Separating them by restoring normal temperature and pressure, refined perfluoropolyether lubricant can be recovered. The carbon dioxide can be recovered and recycled as
20 required.

In the way as describe above, perfluoropolyether lubricant can be refined removing ionic impurities from the lubricant. The above-mentioned lubricants available from Ausimont KK can be refined by

removing ionic impurities of sodium ions, potassium ions, chloride ions, HCO_3 ions, HSO_4 ions, and sulfuric ions.

The refined perfluoropolyether lubricant can be used for
5 lubricant in a magnetic recording medium according to the invention as described later. Because lubricant refined by this mode of the invention contains reduced amount of ionic impurities, a magnetic recording medium performing excellent long term stability can be provided when such lubricant is used for material of a lubricant layer
10 of the magnetic recording medium.

Next, second mode of the invention will be described in the following. The second mode of the invention is a method for refining perfluoropolyether lubricant removing perfluoropolyether compounds
15 having a terminal group with weak polarity from the lubricant. The second mode refines lubricant by assorting terminal groups of the lubricant. In order to improve adhesion and bonding of a perfluoropolyether lubricant with a protective layer, it is effective to use lubricant predominantly containing such perfluoropolyether
20 compounds that have the terminal group exhibiting high adhesiveness and bonding characteristic with the protective layer. When lubricant includes such perfluoropolyether compounds that have a terminal group with weak polarity, those compounds need to be assorted and removed in order to improve adhesion and bonding

with the protective layer. This mode of invention provides a refinement method in this case. The compounds that can be removed by a method of this mode of the invention include perfluoropolyether compounds having a terminal group such as CF_3 , CF_2Cl , or CF_2H .

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The second mode of the invention is specifically described below. The second mode of the invention is a method for refining perfluoropolyether lubricant by means of supercritical extraction, wherein the lubricant is contacted with extracting medium of
10 supercritical carbon dioxide under a predetermined condition in a pressure vessel to remove perfluoropolyether compounds exhibiting weak polarity from the lubricant. More specifically, this mode of refinement method comprises following steps.

- 15 (a) A step of introducing perfluoropolyether lubricant into a pressure vessel,
(b) a step of contacting the lubricant with supercritical carbon dioxide under a predetermined condition in the pressure vessel to extract a perfluoropolyether compounds having a terminal group with weak
20 polarity, and
(c) a step of recovering the perfluoropolyether compounds remained after the step (b).

This mode is described below following these steps.

Step (a) is the same as in the first mode described above. The vessel and the lubricant to be refined are also the same as in the first mode.

Step (b)

5 The lubricant introduced into the pressure vessel is contacted with the extracting medium of supercritical carbon dioxide, to extract and remove perfluoropolyether compounds having a terminal group with weak polarity.

10 The extracting medium is supercritical carbon dioxide. The supercritical carbon dioxide flows through the pressure vessel at a predetermined rate and contacts with the raw lubricant in the vessel.

While the step (b) can be conducted in the temperature and
15 pressure range corresponding to the density range of 0.4 to 0.9 g/cm³ as mentioned previously, the step is preferably conducted within confined temperature and pressure in that range. The preferable temperature and pressure in conformity with this mode of the invention are the temperature and pressure that allow the state with
20 the density of the supercritical carbon dioxide of not larger than about 0.6 g/cm³. Specific examples of the boundary temperature and pressure can be 60°C, 16 MPa, and 100°C, 22 MPa. By contacting the lubricant with the supercritical carbon dioxide under such a condition, perfluoropolyether compounds having a terminal group with weak

polarity are selectively extracted and removed. Refined lubricant remains in the vessel.

A flow rate of the supercritical carbon dioxide in this step varies
5 with the extraction condition and a target compound to be extracted,
and is not limited to a special value. However, a flow rate that allows
enough contact between the lubricant and the supercritical carbon
dioxide is desired. A preferable flow rate is in the range from 1
mL/min to 100 mL/min per 100 g of the lubricant, more preferably,
10 from 10 mL/min to 50 mL/min per 100 g of the lubricant. Excessively
low flow rate causes a problem of low processing speed. Excessively
high flow rate causes decrease of extraction efficiency and raises a
problem of unnecessary consumption and a problem that the
lubricant is driven out unless the vessel volume is increased.

15

Step (c).

The perfluoropolyether lubricant remained in the vessel is
recovered. The perfluoropolyether lubricant is recovered by stopping
supply of the supercritical carbon dioxide and returning the state in
20 the vessel to the normal temperature and pressure.

In the way as described above, perfluoropolyether lubricant can
be refined removing perfluoropolyether compounds with weak
polarity from the lubricant.

When the raw lubricant mentioned in the first mode above is used, the perfluoropolyether lubricant refined by this second mode mainly includes the perfluoropolyether compounds having highly polar terminal group, such as piperonyl group or hydroxyl group.

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The refined perfluoropolyether lubricant can be used for lubricant in a magnetic recording medium according to the invention as described later. Because lubricant refined by this mode contains reduced amount of perfluoropolyether compounds having a weak polarity terminal group, a magnetic recording medium performing excellent adhesion and bonding of the lubricant can be provided when such lubricant is used for material of a lubricant layer of the magnetic recording medium. Since perfluoropolyether compounds having a weak polarity terminal group are removed from the lubricant that is refined by this invention, the refined lubricant performs strong adhesion and bonding with a protective layer of a magnetic recording medium as compared with unrefined lubricant having the same molecular weight. Thus, an enough heat resistant lubricant layer can be formed.

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Third mode of the invention will be described in the following. The third mode of the invention is a method for refining perfluoropolyether lubricant removing both perfluoropolyether compounds having a weak polarity terminal group and ionic

impurities from the lubricant. In this mode, a single refinement process can remove both perfluoropolyether compounds having a weak polarity terminal group and ionic impurities; thus a method of this mode performs high productivity.

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The third mode of the invention is specifically described below.

The third mode is a method for refining perfluoropolyether lubricant by means of supercritical extraction, wherein the lubricant is

contacted with extracting medium of supercritical carbon dioxide

10 under a first condition in a pressure vessel to extract and remove perfluoropolyether compounds having a weak polarity terminal group from the lubricant, and then the lubricant from which

perfluoropolyether compounds having a weak polarity terminal group have been removed is contacted with extracting medium of

15 supercritical carbon dioxide under a second condition to extract and recover perfluoropolyether lubricant while removing ionic impurities.

More specifically, this mode of refinement method comprises the following steps.

20 (a) A step of introducing perfluoropolyether lubricant into a pressure vessel,

(b) a step of contacting the lubricant with supercritical carbon dioxide under a first condition in the pressure vessel to extract perfluoropolyether compounds having a weak polarity terminal group,

and

(c) a step of contacting the perfluoropolyether lubricant remained after the step (b) with supercritical carbon dioxide under a second condition to extract and recover the perfluoropolyether lubricant.

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This mode is described below following these steps.

Step (a) is the same as in the first mode described above. The vessel and the lubricant to be refined are also same as in the first mode.

10 Step (b)

The lubricant introduced into the pressure vessel is contacted with the extracting medium of supercritical carbon dioxide under a first condition to extract and remove perfluoropolyether compounds having a weak polarity terminal group. When the raw material is the
15 lubricant described in the first mode above, the perfluoropolyether compounds having a terminal group of CF_3 , CF_2Cl , or CF_2H , for example, are removed.

The extracting medium is supercritical carbon dioxide. The
20 supercritical carbon dioxide flows through the pressure vessel at a predetermined rate and contacts with the raw lubricant in the vessel.

Extraction in this step (b) is conducted under the first condition. While the first condition can be in the temperature and pressure

range corresponding to the density range of 0.4 to 0.9 g/cm³ as mentioned previously, this step is preferably conducted at a temperature and pressure in which density of the supercritical carbon dioxide is not larger than about 0.6 g/cm³. This upper limit of the
5 density corresponds to specific temperature and pressure of , for example, 60°C, 16 MPa, or 100°C, 22 MPa,. By contacting the lubricant with the supercritical carbon dioxide under such a condition, components of perfluoropolyether compound having a weak polarity terminal group are selectively extracted.

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A flow rate of the supercritical carbon dioxide in this step varies with the extraction condition and a target compound to be extracted, and is not limited to a special value. However, a flow rate that allows enough contact between the lubricant and the supercritical carbon
15 dioxide is desired. The preferable flow rate is in the range from 1 mL/min to 100 mL/min per 100 g of the lubricant, more preferably, from 10 mL/min to 50 mL/min per 100 g of the lubricant. Excessively low flow rate causes a problem of low processing speed. Excessively high flow rate causes decrease of extraction efficiency and raises a
20 problem of unnecessary consumption and a problem that the lubricant is driven out unless the vessel volume is increased.

After this step, perfluoropolyether compounds having a high polarity group are remained in the vessel.

Step (c).

The components of the lubricant remained in the vessel are contacted with the extraction medium of supercritical carbon dioxide to extract perfluoropolyether compounds having a terminal group
5 with higher polarity.

The extracting medium is supercritical carbon dioxide. The supercritical carbon dioxide flows through the pressure vessel at a predetermined rate and contacts with the lubricant components in
10 the vessel.

The extraction in this step (c) is conducted under the second condition. While the second condition can be in the temperature and pressure range corresponding to the density range of 0.4 to 0.9 g/cm³
15 as mentioned previously, this step is preferably conducted at a temperature and pressure in which the density of the supercritical carbon dioxide is not larger than about 0.7 g/cm³. This upper limit of the density corresponds to specific temperature and pressure of, for example, 60°C, 20 MPa, or 100°C, 26 MPa. Because the step (c)
20 extracts perfluoropolyether having a terminal group with higher polarity than in the perfluoropolyether extracted in the step (b), an additional condition of the temperature and pressure is preferably held in which the density is higher than the upper limit density in the step (b), as well as the second condition. That is, the condition of

the temperature and pressure in the step (c) are most preferably such that the density of the supercritical carbon dioxide in that condition is from 0.6 g/cm³ to 0.7 g/cm³.

5 By contacting the lubricant with the supercritical carbon dioxide under such a condition, components of perfluoropolyether compounds are selectively extracted remaining ionic impurities in the residue in the vessel.

10 A flow rate of the supercritical carbon dioxide in this step varies with the extraction condition and a target compound to be extracted, and is not limited to a special value. However, a flow rate that allows enough contact between the lubricant and the supercritical carbon dioxide is desired. The preferable flow rate is in the range from 1
15 mL/min to 100 mL/min per 100 g of the lubricant, more preferably, from 10 mL/min to 50 mL/min per 100 g of the lubricant. Excessively low flow rate causes a problem of low processing speed. Excessively high flow rate causes decrease of extraction efficiency and raises a problem of unnecessary consumption and a problem that the
20 lubricant is driven out unless the vessel volume is increased.

Further in the step (c), the extracted perfluoropolyether compounds are collected together with the supercritical carbon dioxide to recover the perfluoropolyether compounds. The

perfluoropolyether compounds and the supercritical carbon dioxide are taken out through an outlet port of the pressure vessel. They are separated by returning to the normal temperature and pressure. Recovering the separated perfluoropolyether compounds, refined
5 perfluoropolyether lubricant is obtained. The carbon dioxide can be recovered and recycled as required.

As described above, lubricant can be refined by removing ionic impurities and perfluoropolyether compounds having a weak polarity
10 terminal group from perfluoropolyether lubricant.

The perfluoropolyether lubricant refined by this mode of the invention, when the raw lubricant described in the first mode above is used, mainly includes the perfluoropolyether compounds having a
15 terminal group with higher polarity, such as a piperonyl group or a hydroxyl group. At the same time, ionic impurities including sodium ions, potassium ions, chloride ions, HCO_3 ions, HSO_4 ions, and sulfuric ions have been removed from the refined lubricant.

20 The refined perfluoropolyether lubricant can be used for lubricant in a magnetic recording medium according to the invention as described later. Because the lubricant refined by this mode contains reduced amount of perfluoropolyether compounds having a weak polarity terminal group, and ionic impurities are also removed

from the refined lubricant, when used for material of a lubricant layer, a magnetic recording medium can be provided that performs excellent adhesion and bonding of the lubricant, and thus superior heat resistance, and besides, long term stability.

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The refinement method of the invention refines lubricant not by a separation means such as a column using packing materials.

Consequently, the method is inexpensive, superior in durability, and as a result, achieves high productivity. In particular, the method of
10 the invention is versatile and applicable to refine from small to large amounts of lubricant as described later in the examples of embodiment. The method of the present invention allows refining lubricant based on the difference in molecular weight or a functional group of perfluoropolyether, or little difference in polarity of
15 compounds and ions contained in the lubricant. Besides, the refinement method of the invention may be applied to both a batch system and a continuous system by appropriate selection of a vessel and other items.

20 The second invention will be described in the following. The second invention is lubricant that is refined by a method of the first invention described above, and in particular, lubricant used for a magnetic recording medium. Specifically, the lubricant of the invention is lubricant refined removing ionic impurities, lubricant

refined removing perfluoropolyether components having a weak polarity terminal group, or lubricant refined by removing both ionic impurities and perfluoropolyether components having a weak polarity terminal group.

5

Lubricant according to the present invention is lubricant with increased content of perfluoropolyether compounds having a terminal group with high polarity by sorting perfluoropolyether compounds based on the difference in polarity between terminal groups of perfluoropolyether molecules and the difference in polarity between ionic impurities and perfluoropolyether compounds. Consequently, adhesiveness and bonding strength of the lubricant with a protective layer is enhanced, and as a result, spin migration is suppressed and vaporization of the lubricant by heat is prevented.

15

Because ionic impurities that cause decomposition of the lubricant are eliminated in the lubricant of the invention, the decomposition of the lubricant is suppressed, assuring long term stability. Ionic impurities that are contained in unrefined lubricant mentioned in the description of the first invention may be Na^+ , K^+ , Cl^- , HCO_3^- , SO_4^{2-} , or HSO_4^- .

The third invention will be described in the following. The third invention is a magnetic recording medium using lubricant according

to the present invention described above.

The third invention will be described below referring to the accompanying drawing. Though the following describes an aspect of
5 embodiment of the invention, the present invention shall not be limited to the aspect of embodiment.

Fig. 1 is a schematic cross sectional view of a magnetic recording medium according to the present invention. As shown in Fig. 1, a
10 magnetic recording medium of the invention comprises a nonmagnetic underlayer 3, a magnetic recording layer 4, a protective layer 5, and a lubricant layer 6 sequentially laminated on a substrate.

As shown in Fig. 1, a substrate in the invented medium
15 preferably comprises a nonmagnetic base plate 1 and a plating layer 2, a nickel-phosphorus (Ni-P) plating layer, for example, formed on the base plate by electroless plating. (The combination of the nonmagnetic base plate 1 and the plating layer 2 is called a substrate in this specification.)

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The nonmagnetic base plate 1 is formed with a material used in a conventional magnetic recording medium. For example, material used for the base plate 1 may be an aluminum alloy, strengthened glass, crystallized glass, or plastic.

A nonmagnetic underlayer 3, a magnetic layer 4, a protective layer 5, and a lubricant layer 6 are sequentially formed on the substrate to produce a magnetic recording medium.

5 Conventional materials can be used for the nonmagnetic underlayer 3, a magnetic layer 4, and the protective layer 5. Specifically, the nonmagnetic underlayer 3 can be an underlayer composed of chromium; the magnetic layer 4 can be composed of a cobalt alloy, for example, a ferromagnetic alloy of Co-Cr-Ta or Co-Cr-
10 Pt; and the protective layer 5 can be a carbon protective layer.

The lubricant layer 6 is essentially composed of perfluoropolyether lubricant that is refined by a refinement method according to the present invention.

15

Although a magnetic recording medium according to the present invention is described above referring to Fig. 1, the structure of Fig. 1 is only an example of a structure of a magnetic recording medium according to the invention and variations are possible complying with
20 each object of the recording medium. A shape of the medium can be adapted for a device that uses the medium, and is not limited to a special shape. An example may be a disk shape magnetic recording medium mounted on a HDD.

A manufacturing process of a magnetic recording medium of the invention includes steps of coating the substrate described above with a nonmagnetic underlayer 3 and sequentially forming a magnetic layer 4 and a protective layer 5 on the underlayer. After that,
5 lubricant of the present invention is applied to the surface of the protective layer 5.

The nonmagnetic underlayer 3 is preferably a chromium layer, and the magnetic layer 4 is preferably a cobalt alloy layer in the
10 present invention.

The nonmagnetic underlayer 3, the magnetic layer 4, and the protective film 5 can be formed by means of a DC sputtering method when the three layers are a chromium nonmagnetic underlayer, a
15 cobalt magnetic alloy layer, and a carbon protective layer, respectively. The carbon protective layer may be either a usual carbon protective layer mainly composed of graphite or a DLC protective layer. The lubricant layer 6 can be applied by means of dip-coating or spin-coating. For thicknesses and deposition
20 conditions of the nonmagnetic underlayer 3, the magnetic layer 4, the protective layer 5, and the lubricant layer 6, the same conditions as in usual magnetic recording media may be employed. The construction described above shall not limit the present invention.

Examples

The present invention will be described more in detail referring to examples of preferred embodiment in the following. The present invention, however, shall not be limited by those examples.

- 5 Examples of perfluoropolyether lubricant in the following examples of preferred embodiment are “Fomblin Z tetraol (also simply called as Z-tetraol)” and “AM3001” commercially available from Ausimont KK.

Example 1

- 10 Forty grams of lubricant Z-tetraol was placed in an extraction vessel that is a stainless steel vessel with thick wall provided with inlet and outlet ports, having a content volume of 80 mL.

- Supercritical carbon dioxide was flown through the vessel at the flow rate of 8 mL/min. Temperature was fixed at 60°C and pressure was
15 changed with time. The extraction components of from ZT60-1 to ZT60-10 as shown in Table 1 were obtained at each fraction of the pressure level. Table 1 also shows the pressure and the passage of time.

20

Table 1 Extraction of Z-tetraol at 60°C

sample	pressure (MPa)	time (min)	mass (g)
ZT60-1	10.0→ 11.0→ 11.5 → 12.0→ 12.5→ 13.0	7→ 5→ 5→ 5→ 5 → 20	3.51
ZT60-2	14.0	20	4.12
ZT60-3	15.0	20	5.38
ZT60-4	16.0	20	7.27
ZT60-5	17.0	20	6.40
ZT60-6	18.0	20	4.31
ZT60-7	19.0→ 20.0	10→ 10	3.01
ZT60-8	22.0→ 24.0→ 26.0	10→ 5→ 5	2.25
ZT60-9	28.0→ 30.0	10→ 20	2.25
ZT60-10	residue	-	1.03
		total mass	39.53

Example 2

5 Forty grams of the lubricant (Z-tetraol) was placed in the same vessel as in Example 1 and the supercritical carbon dioxide was flown at the same flow rate. Temperature was fixed at 100°C and pressure was changed with time. The extraction components of from ZT100-1 to ZT100-11 as shown in Table 2 were obtained at each fraction of the
10 pressure level. Table 2 also shows the pressure and the passage of time.

Table 2 Extraction of Z-tetraol at 100°C

sample	pressure (MPa)	time (min)	mass (g)
ZT100-1	10.0→ 11.0→ 12.0→ 13.0→ 14.0→ 15.0→ 16.0→ 17.0→ 18.0	5→ 5→ 5→ 5→ 5 → 5→ 5→ 5→ 5	0.78
ZT100-2	19.0	20	2.31
ZT100-3	20.0	20	3.32
ZT100-4	21.0	20	4.24
ZT100-5	22.0	20	6.27
ZT100-6	23.0	20	7.32
ZT100-7	24.0	20	6.80
ZT100-8	25.0	20	3.41
ZT100-9	26.0	20	1.76
ZT100-10	27.0→ 30.0	3→ 20	1.45
ZT100-11	residue	-	0.71
		total mass	38.36

Example 3

5 Forty grams of the lubricant AM3001 was placed in the same vessel as in Example 1 and the supercritical carbon dioxide was flown at the same flow rate. Temperature was fixed at 60°C and pressure was changed with time. The extraction components of from AM60-1 to AM60-3 as shown in Table 3 were obtained at each fraction of the pressure level. Table 3 also shows the pressure and the passage of time.

10

Table 3 Extraction of AM3001 at 60°C

sample	pressure (MPa)	time (min)	mass (g)
AM60-1	13.0	40	16.5
AM60-2	19.0	30	16.3
AM60-3	residue	-	6.0
		total mass	38.8

Example 4

5 Forty grams of the lubricant AM3001 was placed in the same vessel as in Example 1 and the supercritical carbon dioxide was flown at the same flow rate. Temperature was fixed at 65°C and pressure was changed with time. The extraction components of from AM65-1 to AM65-3 as shown in Table 4 were obtained at each fraction of the
10 pressure level. Table 4 also shows the pressure and the passage of time.

Table 4 Extraction of AM3001 at 65°C

sample	pressure (MPa)	time (min)	mass (g)
AM65-1	13.0	40	12.0
AM65-2	19.0	30	15.5
AM65-3	residue	-	12.3
		total mass	39.8

15

Example 5

250 g of lubricant AM3001 was placed in an extraction vessel that is a stainless steel vessel with thick wall provided with inlet and outlet ports, having a content volume of 450 mL. Supercritical carbon
5 dioxide was flown through the vessel at the flow rate of 20 mL/min. Temperature was fixed at 63°C and pressure was held at 14.5 MPa for 60 min. 100 g of the fraction extracted during this period was removed. After that, the pressure was increased to 16 MPa and held for 40 min to obtain 100 g of target component. Molecular weight of
10 this target component was measured by means of GPC method to obtain the number average molecular weight M_n of 3,400 and the weight average molecular weight M_w of 3,900.

Content of ionic impurities of the sample before and after the
15 refinement process was measured by means of ion chromatography. The sample after the refinement process is the above-mentioned target component. Resulted content of sodium ions decreased from 1.4 ppm to 0.3 ppm, potassium ions decreased from 1.0 ppm to 0.05 ppm, HSO_4 ions decreased from 0.03 ppm to below detection limit of
20 0.01 ppm, chloride ions decreased from 0.07 ppm to 0.04 ppm. Ammonium ions, nitric ions, formic ions, and oxalic ions were scarcely detected in the both samples before and after the refinement process.

Comparative Example 1

The commercial lubricant AM3001 as supplied and not refined was used as the Comparative Example 1 for the following tests.

5 Comparative Example 2

Forty grams of the lubricant AM3001 and 40 g of butyl acetate were mixed and stirred at 60°C (fixed) for 30 min. After standing for 12 hr, the lower portion of the mixture was separated by a separatory funnel. Butyl acetate remaining in the lower portion was eliminated
10 by an evaporator to obtain 20 g of refined lubricant (hereinafter called as “butyl acetate-refined AM”).

Evaluation

1. Analysis results by means of time of flight secondary ion mass
15 spectroscopy (TOF-SIMS)

A few micrograms of each of extraction fractions from Example 1 and Example 2, and a sample of Comparative Example 1 was dropped on the surface of a magnetic recording medium not coated with
20 lubricant. Analysis was conducted on the surfaces by means of TOF-SIMS. The results are shown in Table 5. Table 5 shows the fragment of anions and cations out of various types of fragments detected in the extracted fractions and the sample. Each figure in the Table indicates quantity of the fragments and is number of counts per unit

time detected by the TOF-SIMS.

Table 5 Detected ions by TOF-SIMS

component	cation		anion		
	K	Na	Cl	HCO ₃	HSO ₄
unrefined ZT	45684	925	1793	57	462
ZT60-1	304	56	3110	11	20
ZT60-2	220	42	2040	3	39
ZT60-3	171	20	1381	5	19
ZT60-4	136	14	289	3	24
ZT60-5	214	24	144	14	36
ZT60-6	425	22	259	4	34
ZT60-7	1211	51	193	9	21
ZT60-8	6154	190	264	7	42
ZT60-9	7157	333	519	15	74
ZT60-10	107611	3269	2236	115	353
ZT100-1	7261	215	5399	15	79
ZT100-2	736	40	3544	5	18
ZT100-3	350	16	2624	4	17
ZT100-4	433	32	2154	7	25
ZT100-5	552	20	937	2	23
ZT100-6	531	15	340	4	27
ZT100-7	349	7	263	0	25
ZT100-8	387	19	261	5	25
ZT100-9	1563	28	204	6	21
ZT100-10	4574	69	455	12	40
ZT100-11	102855	4584	2060	161	452

5 As Table 5 shows, the fragments of the cations were detected much in the fractions extracted at high pressures and the residue after extraction. The fragments of the anions HCO₃ and HSO₄ were detected much in the residue after extraction. The fragments of the

anion Cl were detected in the fractions extracted at low pressures (ZT60-1 to ZT60-3 and ZT100-1 to ZT100-4) in addition to in the fraction extracted at high pressure. In the fractions of low pressure extraction ZT60-1 to ZT60-3 and ZT100-1 to ZT100-4, the fragment of
5 CF₂Cl was detected as well as the fragment of Cl. In contrast, in the fractions of high pressure extraction ZT60-9 and in the residues ZT60-10 and ZT100-11, the fragment of CF₂Cl was not detected. Therefore, the Cl fragments in ZT60-9, ZT60-10, and ZT100-11 are attributable to chloride ions, while the Cl fragments in ZT60-1 to
10 ZT60-3 and ZT100-1 to ZT100-4 are originated from lubricant compounds including CF₂Cl.

The above results show that the extraction with carbon dioxide at the temperature of 60°C and pressure higher than 20 MPa involves
15 extraction of accompanied sodium ions, potassium ions, chloride ions, and HSO₄ ions, and causes unfavorable condition for lubricant. Likewise, the condition at the temperature of 100°C and pressure higher than 26 MPa also causes similar result. Carbon dioxides at these two boundary states of temperature and pressure have the
20 same density 0.7 g/cm³. It can be considered that supercritical carbon dioxide having a density higher than this value dissolves ionic impurities contained in perfluoropolyether lubricant. Therefore, in order to remove ionic impurities of perfluoropolyether lubricant, supercritical extraction is preferably conducted using supercritical

carbon dioxide under a condition corresponding to a density not higher than about 0.7 g/cm³ that is the density at 60 °C, 20 MPa and 100 °C, 26 MPa.

5 The behavior of Cl fragments shows that low pressure extraction fractions contain a weak polarity perfluoropolyether compound having a terminal group of CF₂Cl that is a weak polarity group. Consequently, it may be considered that weak polarity perfluoropolyether compounds are abundantly contained in the
10 fractions extracted at a condition corresponding to a density lower than about 0.6 g/cm³ that is the density at 60 °C, 16 MPa and 100 °C, 22 MPa. Therefore, in order to remove weak polarity perfluoropolyether compounds of perfluoropolyether lubricant, supercritical extraction is preferably conducted using supercritical
15 carbon dioxide under a condition corresponding to a density lower than the density at 60 °C, 16 MPa, or at 100 °C, 22 MPa. The boundary density is about 0.6 g/cm³.

These results can be summarized as follows.

20 In order to remove ionic impurities, supercritical extraction is conducted using supercritical carbon dioxide under a condition corresponding to a density not higher than the density at 60 °C, 20 MPa or 100 °C, 26 MPa. The density at the boundary condition is about 0.7 g/cm³.

In order to reduce content of weak polarity perfluoropolyether compounds, supercritical extraction is conducted using supercritical carbon dioxide under a condition corresponding to a density not higher than the density at 60 °C, 16 MPa, or at 100 °C, 22 MPa. The
5 boundary density is about 0.6 g/cm³.

In order to obtain perfluoropolyether lubricant from which both ionic impurities and weak polarity perfluoropolyether compounds are removed, supercritical extraction is conducted using supercritical
10 carbon dioxide, at first, under a condition corresponding to a density not higher than the density at 60 °C, 16 MPa, or at 100 °C, 22 MPa. The boundary density is about 0.6 g/cm³. Subsequently, supercritical extraction is conducted using supercritical carbon dioxide under a condition corresponding to a density not higher than the density at 60
15 °C, 20 MPa or 100 °C, 26 MPa. The density at the boundary condition is about 0.7 g/cm³.

The above-described extraction is possible because firstly, the supercritical carbon dioxide is a non-polar solvent, and secondly,
20 density of the supercritical carbon dioxide continuously changes with temperature and pressure from 0.4 g/cm³ to 0.9 g/cm³, and dissolving ability also increases accompanied by the increase in density. Accordingly, ionic impurities not exemplified above can also be separated taking advantage of difference between solubility of a

target lubricant component and solubility of an ionic impurity in the supercritical carbon dioxide. Thus, metal ions, ammonium ions, and organic ions such as oxalic ions and formic ions can be separated.

5 2. Measurement of lubricant properties

Lubricant properties have been measured by molecular weight measurement, supercritical fluid chromatography (SFC), measurement of the temperature at 10 % weight loss of lubricant by means of thermo gravimetric analysis (TG), and a spin-off test on a
10 magnetic recording medium.

(i) Molecular weight measurement

Measurement of molecular weight was conducted on the samples of AM65-2 of Example 4, AM60-2 of Example 3, unrefined AM of
15 Comparative Example 1, and butyl acetate-refined AM of Comparative Example 2. The measurement of molecular weight was performed by means of known GPC method to obtain number average molecular weight M_n and weight average molecular weight M_w . Results are shown in Table 6.

20

(ii) Measurement of terminal groups by means of supercritical fluid chromatography (SFC)

The lubricant fraction AM65-2 of Example 4 was subjected to SFC. Conditions of the SFC were as follows.

Mobile phase: CO₂ at 40 °C, 20 MPa, flow rate: 3 mL/min

Injected mass of the sample: 5 µg

Detector: ELSD

A silica gel column was used.

5

The chromatogram shown in Fig. 2 was obtained. The fraction corresponding to the first peak of the chromatogram was separated and subjected to analysis by means of TOF-SIMS. The separated fraction intensely showed the fragments of CF₂Cl and CF₂H and very weakly showed the fragment based on piperonyl group that is a terminal group of a high polar perfluoropolyether compound.

10

Consequently, the first peak appeared in the SFC can be attributed to lubricant molecules having a weak polarity terminal group. Since the CF₃ fragment in the TOF-SIMS develops from the principal chain of the lubricant as well, existence of a lubricant component having a terminal group of CF₃ is unclear. However, if such a component exists, it must be contained in the first peak because of the property of SFC. Areal proportion of the first peak with respect to the total peaks was calculated on the AM65-2.

15

20

Areal proportion of the first peak with respect to the total peaks was calculated on the samples of AM60-2 of Example 3, unrefined AM of Comparative Example 1, and butyl acetate-refined AM of Comparative Example 2, as well as on the AM65-2 of Example 4.

- (iii) Measurement of the temperature at 10 % weight loss of lubricant by means of thermo gravimetric analysis (TG)

Analysis by TG method was conducted to measure weight loss of lubricant due to heating. While heating each sample, the temperature when the weight of the lubricant decreased by 10 % was measured by the TG method and recorded. The measurements were conducted on the samples of AM65-2 of Example 4, AM60-2 of Example 3, unrefined AM of Comparative Example 1, and butyl acetate-refined AM of Comparative Example 2. The results are shown in Table 6.

- (iv) Spin-off test of magnetic recording media

Spin-off tests were conducted on the samples of AM65-2 of Example 4, AM60-2 of Example 3, unrefined AM of Comparative Example 1, and butyl acetate-refined AM of Comparative Example 2.

Ni-P plating was executed by electroless plating to form a plating layer on a nonmagnetic base plate of aluminum-magnesium alloy. After polishing the surface, nearly concentric grooves with the surface roughness Ra of 0.8 nm were formed by texturing using diamond slurry. After cleaning, this substrate was put into a commonly used sputtering apparatus, in which formed were a nonmagnetic underlayer of chromium, a magnetic layer comprising Co, Cr, Ta, and

Pt, and a protective layer of nitrogen-doped amorphous carbon (a-C:N) having thickness of 8 nm by a CVD method. Thus, a magnetic recording disk having diameter of 95 mm was obtained. Lubricant was applied by dip-coating on the magnetic recording disk to the thickness of 1.6 nm to complete a magnetic recording medium. Above-mentioned four types of lubricant were used for sample of the lubricant.

The spin-off test was executed by rotating the magnetic recording medium prepared as described above at 7,200 rpm for 120 hr in an environment of the temperature of 55°C and the relative humidity of 80 %. Variation of the film thickness (%) was obtained by measuring the thicknesses at the radius of 18 mm, which is within the CSS region, before and after the rotation test. Results are given in Table 6.

Table 6 Properties of the four types of lubricant

	Mn	Mw	first peak area (*1)	temperature at 10 % loss (*2) (°C)	thickness change after spin- off test
unrefined AM	3,000	3,900	14%	302	-14%
AM60-2	3,500	4,700	6%	334	-4%
AM65-2	3,100	3,600	7%	324	-10%
butyl acetate- refined AM	3,900	4,800	18%	329	-10%

(*1) areal proportion of the first peak in the SFC

(*2) temperature at 10 % weight loss of lubricant in TG analysis

5

As Table 6 shows, the areal proportion of the first peak in the SFC of AM60-2 and AM65-2 that were refined by the method of the invention was reduced to half or lower than the proportion of the unrefined AM. The result confirms reduction of components of lubricant molecules having a terminal group of weak polarity.

10

The molecular weights of AM60-2 and AM65-2 were smaller than that of butyl acetate-refined AM, while the temperature of 10 % loss of the lubricant and the spin-off test result were nearly the same as in the butyl acetate-refined AM. Accordingly, the lubricant refined by the method of the invention has high bonding property despite lower molecular weight. This is caused by removal of components of lubricant having a weak polarity terminal group.

15

A magnetic recording medium using lubricant refined by the method of the invention has relatively small effect on a magnetic head even with small flying height of the head. Therefore, a magnetic recording medium using lubricant of the invention performs
5 superior long term stability.

Advantageous Effect of the invention

A method for refining lubricant according to the present invention removes either one or both of ionic impurities and
10 perfluoropolyether components having a weak polarity terminal group by controlling a state of carbon dioxide used in supercritical-fluid extraction. Thus, a refinement method with high productivity is provided. The present invention also provides lubricant exhibiting high bonding performance despite low molecular weight by employing
15 the refining method. Further, lubrication performance of a magnetic recording medium in a fixed magnetic recording apparatus is improved by using the lubricant refined by the invented method. Thus, a magnetic recording medium performing excellent long term stability has been provided.

20

Claims

1. A method for refining lubricant containing a compound having perfluoropolyether structure by means of supercritical extraction, wherein the lubricant is contacted with extracting
5 medium of supercritical carbon dioxide under a predetermined condition in a pressure vessel to remove ionic impurities from the lubricant.
2. A method for refining lubricant according to claim 1, wherein the predetermined condition is a combination of a temperature and a
10 pressure at which a density of the supercritical carbon dioxide is not larger than the density at the temperature of 60°C and the pressure of 20 MPa.
3. A method for refining lubricant according to claim 1, wherein the ionic impurities are included in the group consisting of sodium
15 ions, potassium ions, chloride ions, HCO_3 ions, HSO_4 ions, and sulfuric ions.
4. A method for refining lubricant containing a compound having perfluoropolyether structure by means of supercritical extraction, wherein the lubricant is contacted with extracting
20 medium of supercritical carbon dioxide under a predetermined condition in a pressure vessel to remove a perfluoropolyether compound having a terminal group of weak polarity from the lubricant.
5. A method for refining lubricant according to claim 4, wherein

the predetermined condition is a combination of a temperature and a pressure at which a density of the supercritical carbon dioxide is not larger than the density at the temperature of 60°C and the pressure of 16 MPa.

5 6. A method for refining lubricant according to claim 4, wherein the perfluoropolyether compound having a terminal group of weak polarity is a perfluoropolyether compound having a terminal group included in the group consisting of CF₃, CF₂H, and CF₂Cl.

7. A method for refining lubricant containing a compound
10 having perfluoropolyether structure by means of supercritical extraction, wherein the lubricant is contacted with extracting medium of supercritical carbon dioxide under a first condition in a pressure vessel to remove a perfluoropolyether compound having a terminal group of weak polarity from the lubricant, and then the
15 lubricant from which a perfluoropolyether compound having a terminal group of weak polarity is removed is contacted with extracting medium of supercritical carbon dioxide under a second condition to remove ionic impurities from the lubricant and to extract and recover perfluoropolyether lubricant.

20 8. A method for refining lubricant according to claim 7, wherein the first condition is a combination of a temperature and a pressure at which a density of the supercritical carbon dioxide is not larger than the density at the temperature of 60°C and the pressure of 16 MPa, and the second condition is a combination of a temperature and

a pressure at which a density of the supercritical carbon dioxide is not larger than the density at the temperature of 60°C and the pressure of 20MPa.

9. A method for refining lubricant according to claim 7, wherein
5 the perfluoropolyether compound having a terminal group of weak polarity has a terminal group included in the group consisting of CF₃, CF₂H, and CF₂Cl, and the ionic impurities are included in the group consisting of sodium ions, potassium ions, chloride ions, HCO₃ ions, HSO₄ ions, and sulfuric ions.

10 10. A perfluoropolyether lubricant refined by a method defined by any one of claims 1, 4, and 7.

11. A magnetic recording medium comprising a nonmagnetic substrate and layers sequentially laminated on the substrate, the layers including a nonmagnetic underlayer, a magnetic layer, a
15 protective layer, and a lubricant layer, wherein the lubricant layer is essentially composed of a perfluoropolyether lubricant defined by claim 10.