

9609992-4

# VAPOR LUBRICATION OF FRACTIONATED LUBRICANT ON THIN FILM DISCS

The technical field of the present invention is the lubrication of discs for disc drive systems.

Magnetic disc drive systems frequently use the contact start stop (CSS) system. In the CSS system, a recording-reproducing head slider contacts with the surface of a magnetic storage disc in the stand-by condition where the disc is stopped. Conversely, an air gap is formed between the head slider and the surface of the disc, upon rotation of the disc at a present speed, to disengage the head slider from the disc during the recording or reproducing operation. During operation, the disc and the head slider may be abraded or wear at the time the head slider moves away from the surface of the disc, or the head slider accidentally contacts the surface of the rotating disc during the recording or reproducing operation, instead of being maintained in a position floating above the surface of the disc. Contact during rotation of the disc can lead to scratching or other damage of the disc and the head slider.

Some protection of the magnetic recording layer is obtained by an overcoat, where various materials have been reported for use as overcoats. For example, U.S. Patent No. 4,069,360 uses an amorphous inorganic oxide layer formed on the thin film of a magnetic metal medium and a silicon-containing lubricant layer coated over the amorphous inorganic oxide layer. A silicon based oil or silicon type surface coupling agent serves as the lubricant which adheres to the inorganic outside layer. U.S. Patent No. 4,152,487 employs a non-magnetic alloy disc substrate, a thin film of a magnetic metal medium applied on the substrate and a polysilicate layer to cover the magnetic

This Sheet Has Been Replaced



| REPLACED SHEETS                                 |                              | SG |
|-------------------------------------------------|------------------------------|----|
| <input checked="" type="checkbox"/> Description | : paged 1 to paged 8         |    |
| <input checked="" type="checkbox"/> Claim(s)    | : paged 9 to paged 10        |    |
| <input type="checkbox"/> Drawing(s)             | : paged ..... to paged ..... |    |
| <input checked="" type="checkbox"/> Abstract    | : paged 11 to paged .....    |    |
| DATE OF RECEIPT                                 |                              |    |

metal medium. Further lubrication may be obtained using fluorinated polyethers. U.S. Patent No. 4,268,556 employs fluorinated polymers having a low molecular weight and containing a functional group at the termini as a lubricant for magnetic recording discs. U.S. Patent No. 4,529,659 uses a lubricant comprising a fluorinated hydrocarbon polymer having a terminal carboxyl group which forms a salt with an aminosilane layer chemically coupled to a silicon oxide layer applied on the magnetic storage medium of a disc. U.S. Patent No. 5,227,211 employs a perfluoropolyether lubricant for coating onto a carbon overcoat.

Japanese patent publication No. 5-234066 teaches the use of perfluoropolyether (average molecular weight 2.5kD), which has been repeatedly extracted with a polar low molecular weight organic solvent, as a lubricant layer for magnetic recording media. Japanese patent publication No. 6-325360 teaches the use of perfluoropolyether as a lubricant on an amorphous carbon film overcoat of a magnetic recording disc. U.S. Patent No. 5,331,487 teaches the use of a lubricant vapor transport system cooperating with a reservoir to maintain lubrication of a magnetic recording disc. Japanese patent publication No. 4-344318 teaches the use of a perfluoropolyether derivative as a lubricant for a magnetic recording disc, with a ratio of a fraction, soluble to solvents, to that, insoluble to solvents, in the lubricating layer of 0.65-1.22. U.S. Patent No. 4,327,139 employs one or a mixture of perfluoropolyether oils. WO 94/08334 discloses a perfluoropolyether with a surface energy of less than 50 ergs/cm<sup>2</sup>. Japanese patent publication No. 6-004852 which uses a perfluoropolyether lubricating layer of  $2 \times 10^{-9}$ m thick.

Thus, the present invention provides methods and devices comprising magnetic recording discs coated with a narrow molecular weight fraction of a terminally

This Sheet Has Been Replaced



functionalized perfluoropolyether having a molecular weight of at least about 1 kD. The coatings are vapor deposited to provide a thickness of at least about 0.5nm. The resulting coating provides protection against stiction for extended periods of time.

In accordance with the present invention, methods and devices are provided for protecting magnetic recording discs by vapor deposition of a protective layer of a functionalized perfluoropolyether of a predetermined thickness. The perfluoropolyether is of a molecular weight of at least 1 kD, and usually not more than 4 kD, generally in the range of about 1.5 to 3.5 kD, having at least 90 weight percent, preferably at least about 95 weight percent, within a 2 - 3 kD range, preferably within a 2.5 - 3 kD range. The molecular weight range of the fraction will be less than about 4 kD, usually less than about 3 kD, preferably less than about 3 kD and may be in the range of about 0.5 to 2 kD, preferably in the range of about 0.5 to 1.5 kD.

The magnetic recording discs may be varied widely as to the magnetic material, nature of the overcoat, and manner of construction.

The magnetic material may be a cobalt-based alloy e.g., cobalt-chromium/nickel or cobalt-chromium/tantalum/platinum; iron oxides, such as gamma-ferric oxide; etc.

The overcoat or protective coating which is deposited on the magnetic layer may be any one of the common coatings, such as a carbon coating, where the carbon has been applied by vapor deposition, RF plasma or glow discharge, DC magnetron sputtering, or the like, which coating may be doped with hydrogen and/or nitrogen. Other protective coatings include silicates and silicones, polysilanes, silicon carbide, boron nitride, etc.

This Sheet Has Been Replaced



The sliders may be any of the conventional sliders and coated with a variety of coatings compatible with the disc coatings. Slider coatings include diamond-like carbon coated sliders, or other chemically inert thin film.

The polyfluoroether compositions are terminally functionalized with any one of a variety of polar groups, which groups include hydroxyl, carboxy, amino, or the like. These fluorinated oils are commercially available under such tradenames as Fomblin Z-Dol, Fomblin Am2001, Fomblin Z-DISOC (Montedison); Demnum (Daikin) and Krytox (Dupont). The commercially available terminally functionalized perfluoropolyethers are fractionated to provide for relatively narrow molecular weight fractions. Fractionation can be achieved by distillation, chromatography, e.g. HPLC, or other techniques which allows separation by molecular weight.

To provide the lubricating coats, the disc is vapor coated at an elevated temperature, where the perfluorinated polyethers are vaporized and allowed to deposit onto the surface of the disc in an enclosed environment, generally at ambient pressure. Temperatures will generally range from about 150 to 250°C, preferably about 175 to 225°C. Conveniently, 180°C and 200°C are found to provide reasonable deposition times. Deposition times will be at least about 5 min and not more than about thirty hours, preferably not more than about twenty hours. The thickness should be at least about 0.5nm, preferably at least about 1nm, more preferably at least about 1.2nm and will generally be below about 3nm, preferably in the range of about 1 to 3nm, conveniently about 1.5 to 2.5nm. Fractions of particular interest which provide higher film thicknesses are fractions of molecular weight range 2 to 3 kD.

After sufficient time for the desired lubricating layer thickness to be achieved, the magnetic disc is now ready for use. The resulting disc is capable of maintaining a level

This Sheet Has Been Replaced



of stiction up to 20,000 CSS cycles of less than 4g, preferably less than 3g and particularly preferred not more than about 2g.

The following examples are offered in way of illustration and not the way of limitation.

### EXPERIMENTAL

Following the procedure of Coffey et al., IEE Trans. Magn. 30(6), 4146 (1994), magnetic recording discs (glass ceramic substrate, chromium underlayer, cobalt alloy, hydrogenated carbon overcoat) were coated with various fractions obtained from Fomblin Zdol2000. The Fomblin Zdol2000 has a molecular weight range of about 700 to 15,000 Dal, with a number average molecular weight of about 2,000 kDal. The fractions obtained had a narrow molecular weight distribution, with the fraction number indicating increasing molecular weight. The Fomblin Zdol2000 was fractionated by supercritical fluid fractionation. Twenty fractions were obtained. Using the above procedure, the rate of deposition at different temperatures for a single fraction and the film thickness obtained for a number of different fractions at 180°C for 1h were determined. The following tables indicate the results.

---

This Sheet Has Been Replaced



## VAPOR LUBRICATION KINETICS

Lubricant Thickness (nm)

| Temp. °C               | 150 | 180 | 200  |
|------------------------|-----|-----|------|
| Deposition time, hours |     |     |      |
| 0.5                    | 0.5 | 1.7 |      |
| 1                      | 1.2 | 1.8 | 1.9  |
| 5                      | 1.7 | 2   | 2.1* |
| 20                     | 1.9 | 2.5 | 2.7  |

\*Deposition time was 6 hours

## VAPOR LUBRICATION OF FRACTIONATED ZDOL

| Zdol Fraction No. | Film Thickness, nm |
|-------------------|--------------------|
| 2                 | 0.6                |
| 5                 | 0.8                |
| 8                 | 1.1                |
| 9                 | 1.3                |
| 10                | 1.8                |
| 11                | 1.4                |
| 12                | 1.2                |
| 14                | 0.9                |
| 17                | 1.2                |

This Sheet Has Been Replaced



The discs were then tested for stiction using CSS cycles. A strain gauge on an I beam mounted near the head was used to determine stiction. The discs were vapor lubricated at 180°C for one hour using the original composition as obtained from the supplier at a thickness of 0.7 nm, fraction 8 at a thickness of 0.6 nm and fraction 10 at a thickness of 1.6 nm. With no lubrication at all, stiction reached 9g within 2000 cycles. With the original composition, stiction had reached between 6-7g within 2000 cycles. By way of contrast, stiction was constant at 2g for both fractions 8 and 10 out to 20,000 cycles.

It is evident from the above results, that by employing narrow fractions of functionalized perfluoropolyethers, one can achieve stable lubrication over extended periods of time. By vapor deposition, one is able to avoid the necessity of having specific functionalities for covalent bonding to the surface, so as to greatly simplify the manner of lubrication. Uniform coats are obtained, which are substantially maintained during use of the magnetic recording discs.

It should also be evident that such lubricating layer may also, in addition or alternatively, be applied to the head slider.

All publications and patent applications mentioned in this specification are herein incorporated by reference to the same extent as if each individual publication or patent application was specifically and individually indicated to be incorporated by reference.

The invention now being fully described, it will be apparent to one of ordinary skill in the art that many changes and modifications can be made thereto without

This Sheet Has Been Replaced



departing from the scope of the appended claims.

---

This Sheet Has Been Replaced





**CLAIMS:**

1. A disc for use in a disc drive system having a contact start head, said disc comprising a lubricating layer, characterised in that said layer:  
is at least 0.5 nm thick;  
comprises a terminally functionalised perfluoropolyether having a weight average molecular weight in the range of substantially 1 kD to 4 kD; and  
is vapor deposited at an elevated temperature.
2. A disc according to Claim 1, wherein said terminally functionalized perfluoropolyether is functionalized with a member of the group consisting of hydroxyl, carboxy, or amino.
3. A disc according to Claim 1 or 2, wherein said weight average molecular weight is in the range of 2 to 3 kD.
4. A disc according to any one of Claims 1 to 3, wherein said layer is in the range of substantially 1 to 3nm.
5. A disc according to any one of the preceding claims, wherein said disc comprises a magnetic layer, a carbon overcoat deposited there over but beneath said lubricating layer.
6. A disc according to any one of the preceding claims, wherein said elevated temperature is in the range of substantially 150 to 250°C.

This Sheet Has Been Replaced





7. A method of preparing a lubricated disc, said method comprising:  
evaporating at an elevated temperature a terminally functionalized  
perfluoropolyether having a weight average molecular weight in the range of  
substantially 1 to 4 kD, in an enclosed environment containing the disc having a  
magnetic layer for sufficient time for said terminally functionalized perfluoropolyether to  
deposit a layer of at least 0.5nm on said disc.
8. A method according to Claim 7, wherein said terminally functionalized  
perfluoropolyether is functionalized with a member of the group consisting of hydroxyl,  
carboxy, or amino.
9. A method according to Claim 7 or 8, wherein said elevated temperature is in the  
range of substantially 150 to 250 °c.

This Sheet Has Been Replaced



## VAPOR LUBRICATION OF FRACTIONATED LUBRICANT ON THIN FILM DISCS

### ABSTRACT

Lubricated magnetic recording discs are provided by vapor deposition of narrow molecular weight fractions of a terminally functionalized perfluoropolyether. The coatings are found to provide stable lubrication over extended periods of time in CSS cycles.

4-20000000

|                                     |                                          |    |
|-------------------------------------|------------------------------------------|----|
| REPLACEMENT SHEETS                  |                                          | SG |
| <input checked="" type="checkbox"/> | Description : paged ..1.. to paged ..8.. |    |
| <input checked="" type="checkbox"/> | Claim(s) : paged ..9.. to paged ..10..   |    |
| <input type="checkbox"/>            | Drawing(s) : paged ..... to paged .....  |    |
| <input checked="" type="checkbox"/> | Abstract : paged ..11.. to paged .....   |    |
| DATE OF RECEIPT .....12/12/96.....  |                                          |    |

1

9609992-4

VAPOR LUBRICATION OF FRACTIONATED  
LUBRICANT ON THIN FILM DISCS

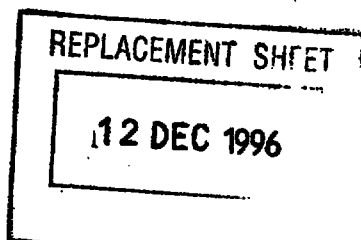
|                   |
|-------------------|
| REPLACEMENT SHEET |
| 12 DEC 1996       |

The technical field of the present invention is the lubrication of discs for disc drive systems.

Magnetic disc drive systems frequently use the contact start stop (CSS) system. In the CSS system, a recording-reproducing head slider contacts with the surface of a magnetic storage disc in the stand-by condition where the disc is stopped. Conversely, an air gap is formed between the head slider and the surface of the disc, upon rotation of the disc at a present speed, to disengage the head slider from the disc during the recording or reproducing operation. During operation, the disc and the head slider may be abraded or wear at the time the head slider moves away from the surface of the disc, or the head slider accidentally contacts the surface of the rotating disc during the recording or reproducing operation, instead of being maintained in a position floating above the surface of the disc. Contact during rotation of the disc can lead to scratching or other damage of the disc and the head slider.

Some protection of the magnetic recording layer is obtained by an overcoat, where various materials have been reported for use as overcoats. For example, U.S. Patent No. 4,069,360 uses an amorphous inorganic oxide layer formed on the thin film of a magnetic metal medium and a silicon-containing lubricant layer coated over the amorphous inorganic oxide layer. A silicon based oil or silicon type surface coupling agent serves as the lubricant which adheres to the inorganic outside layer. U.S. Patent No. 4,152,487 employs a non-magnetic alloy disc substrate, a thin film of a magnetic metal medium applied on the substrate and a polysilicate layer to cover the magnetic

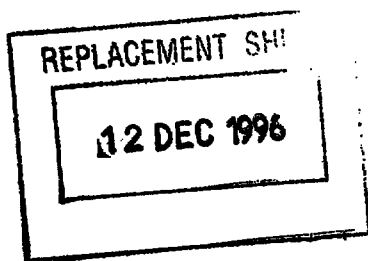
9609992-4



metal medium. Further lubrication may be obtained using fluorinated polyethers. U.S. Patent No. 4,268,556 employs fluorinated polymers having a low molecular weight and containing a functional group at the termini as a lubricant for magnetic recording discs. U.S. Patent No. 4,529,659 uses a lubricant comprising a fluorinated hydrocarbon polymer having a terminal carboxyl group which forms a salt with an aminosilane layer chemically coupled to a silicon oxide layer applied on the magnetic storage medium of a disc. U.S. Patent No. 5,227,211 employs a perfluoropolyether lubricant for coating onto a carbon overcoat.

Japanese patent publication No. 5-234066 teaches the use of perfluoropolyether (average molecular weight 2.5kD), which has been repeatedly extracted with a polar low molecular weight organic solvent, as a lubricant layer for magnetic recording media. Japanese patent publication No. 6-325360 teaches the use of perfluoropolyether as a lubricant on an amorphous carbon film overcoat of a magnetic recording disc. U.S. Patent No. 5,331,487 teaches the use of a lubricant vapor transport system cooperating with a reservoir to maintain lubrication of a magnetic recording disc. Japanese patent publication No. 4-344318 teaches the use of a perfluoropolyether derivative as a lubricant for a magnetic recording disc, with a ratio of a fraction, soluble to solvents, to that, insoluble to solvents, in the lubricating layer of 0.65-1.22. U.S. Patent No. 4,327,139 employs one or a mixture of perfluoropolyether oils. WO 94/08334 discloses a perfluoropolyether with a surface energy of less than 50 ergs/cm<sup>2</sup>. Japanese patent publication No. 6-004852 which uses a perfluoropolyether lubricating layer of  $2 \times 10^{-9}$  m thick.

Thus, the present invention provides methods and devices comprising magnetic recording discs coated with a narrow molecular weight fraction of a terminally



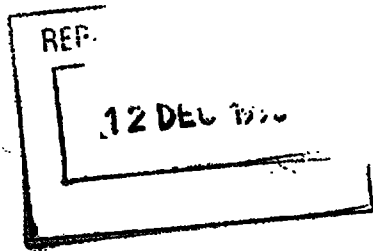
functionalized perfluoropolyether having a molecular weight of at least about 1 kD. The coatings are vapor deposited to provide a thickness of at least about 0.5nm. The resulting coating provides protection against stiction for extended periods of time.

In accordance with the present invention, methods and devices are provided for protecting magnetic recording discs by vapor deposition of a protective layer of a functionalized perfluoropolyether of a predetermined thickness. The perfluoropolyether is of a molecular weight of at least 1 kD, and usually not more than 4 kD, generally in the range of about 1.5 to 3.5 kD, having at least 90 weight percent, preferably at least about 95 weight percent, within a 2 - 3 kD range, preferably within a 2.5 - 3 kD range. The molecular weight range of the fraction will be less than about 4 kD, usually less than about 3 kD, preferably less than about 3 kD and may be in the range of about 0.5 to 2 kD, preferably in the range of about 0.5 to 1.5 kD.

The magnetic recording discs may be varied widely as to the magnetic material, nature of the overcoat, and manner of construction.

The magnetic material may be a cobalt-based alloy e.g., cobalt-chromium/nickel or cobalt-chromium/tantalum/platinum; iron oxides, such as gamma-ferric oxide; etc.

The overcoat or protective coating which is deposited on the magnetic layer may be any one of the common coatings, such as a carbon coating, where the carbon has been applied by vapor deposition, RF plasma or glow discharge, DC magnetron sputtering, or the like, which coating may be doped with hydrogen and/or nitrogen. Other protective coatings include silicates and silicones, polysilanes, silicon carbide, boron nitride, etc.



The sliders may be any of the conventional sliders and coated with a variety of coatings compatible with the disc coatings. Slider coatings include diamond-like carbon coated sliders, or other chemically inert thin film.

The polyfluoroether compositions are terminally functionalized with any one of a variety of polar groups, which groups include hydroxyl, carboxy, amino, or the like. These fluorinated oils are commercially available under such tradenames as Fomblin Z-Dol, Fomblin Am2001, Fomblin Z-DISOC (Montedison); Demnum (Daikin) and Krytox (Dupont). The commercially available terminally functionalized perfluoropolyethers are fractionated to provide for relatively narrow molecular weight fractions. Fractionation can be achieved by distillation, chromatography, e.g. HPLC, or other techniques which allows separation by molecular weight.

To provide the lubricating coats, the disc is vapor coated at an elevated temperature, where the perfluorinated polyethers are vaporized and allowed to deposit onto the surface of the disc in an enclosed environment, generally at ambient pressure. Temperatures will generally range from about 150 to 250°C, preferably about 175 to 225°C. Conveniently, 180°C and 200°C are found to provide reasonable deposition times. Deposition times will be at least about 5 min and not more than about thirty hours, preferably not more than about twenty hours. The thickness should be at least about 0.5nm, preferably at least about 1nm, more preferably at least about 1.2nm and will generally be below about 3nm, preferably in the range of about 1 to 3nm, conveniently about 1.5 to 2.5nm. Fractions of particular interest which provide higher film thicknesses are fractions of molecular weight range 2 to 3 kD.

After sufficient time for the desired lubricating layer thickness to be achieved, the magnetic disc is now ready for use. The resulting disc is capable of maintaining a level

of stiction up to 20,000 CSS cycles of less than 4g, preferably less than 3g and particularly preferred not more than about 2g.

The following examples are offered in way of illustration and not the way of limitation.

### EXPERIMENTAL

Following the procedure of Coffey et al., IEE Trans. Magn. 30(6), 4146 (1994), magnetic recording discs (glass ceramic substrate, chromium underlayer, cobalt alloy, hydrogenated carbon overcoat) were coated with various fractions obtained from Fomblin Zdol2000. The Fomblin Zdol2000 has a molecular weight range of about 700 to 15,000 Dal, with a number average molecular weight of about 2,000 kDal. The fractions obtained had a narrow molecular weight distribution, with the fraction number indicating increasing molecular weight. The Fomblin Zdol2000 was fractionated by supercritical fluid fractionation. Twenty fractions were obtained. Using the above procedure, the rate of deposition at different temperatures for a single fraction and the film thickness obtained for a number of different fractions at 180°C for 1h were determined. The following tables indicate the results.





## VAPOR LUBRICATION KINETICS

Lubricant Thickness (nm)

| Temp. °C<br>Deposition time, hours | 150 | 180 | 200  |
|------------------------------------|-----|-----|------|
| 0.5                                | 0.5 | 1.7 |      |
| 1                                  | 1.2 | 1.8 | 1.9  |
| 5                                  | 1.7 | 2   | 2.1* |
| 20                                 | 1.9 | 2.5 | 2.7  |

\*Deposition time was 6 hours

## VAPOR LUBRICATION OF FRACTIONATED ZDOL

| Zdol Fraction No. | Film Thickness, nm |
|-------------------|--------------------|
| 2                 | 0.6                |
| 5                 | 0.8                |
| 8                 | 1.1                |
| 9                 | 1.3                |
| 10                | 1.8                |
| 11                | 1.4                |
| 12                | 1.2                |
| 14                | 0.9                |
| 17                | 1.2                |



The discs were then tested for stiction using CSS cycles. A strain gauge on an I beam mounted near the head was used to determine stiction. The discs were vapor lubricated at 180°C for one hour using the original composition as obtained from the supplier at a thickness of 0.7 nm, fraction 8 at a thickness of 0.6 nm and fraction 10 at a thickness of 1.6 nm. With no lubrication at all, stiction reached 9g within 2000 cycles. With the original composition, stiction had reached between 6-7g within 2000 cycles. By way of contrast, stiction was constant at 2g for both fractions 8 and 10 out to 20,000 cycles.

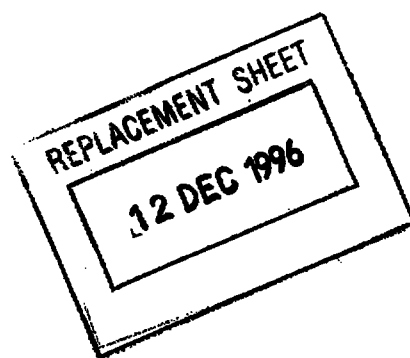
It is evident from the above results, that by employing narrow fractions of functionalized perfluoropolyethers, one can achieve stable lubrication over extended periods of time. By vapor deposition, one is able to avoid the necessity of having specific functionalities for covalent bonding to the surface, so as to greatly simplify the manner of lubrication. Uniform coats are obtained, which are substantially maintained during use of the magnetic recording discs.

It should also be evident that such lubricating layer may also, in addition or alternatively, be applied to the head slider.

All publications and patent applications mentioned in this specification are herein incorporated by reference to the same extent as if each individual publication or patent application was specifically and individually indicated to be incorporated by reference.

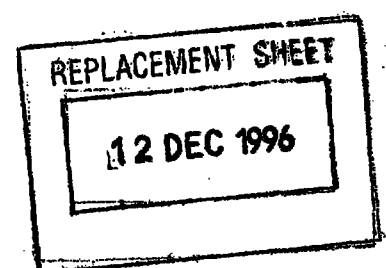
The invention now being fully described, it will be apparent to one of ordinary skill in the art that many changes and modifications can be made thereto without

departing from the scope of the appended claims.



CLAIMS:

1. A disc for use in a disc drive system having a contact start head, said disc comprising a lubricating layer, characterised in that said layer:  
is at least 0.5 nm thick;  
comprises a terminally functionalised perfluoropolyether having a weight average molecular weight in the range of substantially 1 kD to 4 kD; and  
is vapor deposited at an elevated temperature.
2. A disc according to Claim 1, wherein said terminally functionalized perfluoropolyether is functionalized with a member of the group consisting of hydroxyl, carboxy, or amino.
3. A disc according to Claim 1 or 2, wherein said weight average molecular weight is in the range of 2 to 3 kD.
4. A disc according to any one of Claims 1 to 3, wherein said layer is in the range of substantially 1 to 3nm.
5. A disc according to any one of the preceding claims, wherein said disc comprises a magnetic layer, a carbon overcoat deposited there over but beneath said lubricating layer.
6. A disc according to any one of the preceding claims, wherein said elevated temperature is in the range of substantially 150 to 250°C.





7. A method of preparing a lubricated disc, said method comprising:  
evaporating at an elevated temperature a terminally functionalized perfluoropolyether having a weight average molecular weight in the range of substantially 1 to 4 kD, in an enclosed environment containing the disc having a magnetic layer for sufficient time for said terminally functionalized perfluoropolyether to deposit a layer of at least 0.5nm on said disc.
8. A method according to Claim 7, wherein said terminally functionalized perfluoropolyether is functionalized with a member of the group consisting of hydroxyl, carboxy, or amino.
9. A method according to Claim 7 or 8, wherein said elevated temperature is in the range of substantially 150 to 250 °c.



**VAPOR LUBRICATION OF FRACTIONATED  
LUBRICANT ON THIN FILM DISCS**

**ABSTRACT**

Lubricated magnetic recording discs are provided by vapor deposition of narrow molecular weight fractions of a terminally functionalized perfluoropolyether. The coatings are found to provide stable lubrication over extended periods of time in CSS cycles.



4-5888888