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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: POLISHING SLURRY, PRODUCTION METHOD OF GLASS SUBSTRATE FOR INFORMATION RECORDING MEDIUM AND PRODUCTION METHOD OF INFORMATION RECORDING MEDIUM

(57) Abstract: The present invention provides a low-cost polishing slurry having excellent effect with respect to defects and smoothness of the surface to be polished. The polishing slurry comprises a silica abrasive and a ceria abrasive, wherein the silica abrasive content is less than 3 mass% and the ceria abrasive content is less than 1 mass%, based on the entire polishing slurry. Further, the present invention provides a method for producing a crystallized glass substrate for an information recording medium, wherein the method use a polishing slurry of the present invention. Furthermore, the present invention provides a method for producing an information recording medium, comprising forming a recording layer on a crystallized glass substrate for an information recording medium obtained by the present method.



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DESCRIPTION

POLISHING SLURRY, PRODUCTION METHOD OF GLASS SUBSTRATE
FOR INFORMATION RECORDING MEDIUM AND
5 PRODUCTION METHOD OF INFORMATION RECORDING MEDIUM

CROSS REFERENCE TO RELATED APPLICATION

This application is an application filed under 35
U.S.C. § 111(a) claiming benefit pursuant to 35 U.S.C. §
10 119(e) of the filing date of Provisional Application
60/607596, filed on September 8, 2004, pursuant to 35
U.S.C. § 111(b).

TECHNICAL FIELD

The present invention relates to a polishing slurry
15 used for polishing a substrate, a production method of a
glass substrate for an information recording medium, and
a production method of an information recording medium.
RELATED ART

As for an information medium, a magnetic disc, a
20 magneto-optical disc and an optical disc have been used.
Among these, a magnetic disc is used as a high-capacity
information recording medium. The substrate used for
this magnetic disc is an aluminum alloy substrate or a
glass substrate. In the case of using glass-substrate,
25 as the substrate of a magnetic disc, hardness, surface
smoothness, rigidity and impact resistance superior to
those of an aluminum alloy substrate are generally
ensured.

The glass substrate includes a chemically tempered
30 glass substrate and a crystallized glass substrate, but a
crystallized glass substrate has recently attracted
attention because of its high hardness, high rigidity and
high impact resistance. The crystallized glass substrate
comprises a crystalline phase moiety and an amorphous
35 phase moiety. Accordingly, in the polishing of the
surface of the crystallized glass substrate, the
relatively soft amorphous phase moiety is preferentially

polished, the crystalline phase moiety tends to remain and, as a result, high surface smoothness cannot be obtained.

For example, in the case where ceria (cerium oxide: CeO_2) used in the precision polishing of an amorphous glass, such as chemically tempered glass, is used for the polishing of a crystallized glass, the polishing rate greatly differs between the relatively hard crystalline phase moiety and the relatively soft amorphous phase moiety, and the crystalline phase moiety remains to form fine protrusions or depressions on the polished surface, resulting in failure to obtain high smoothness.

Also, in the case of using an aluminum oxide as the abrasive, the abrasive grain is hard and has a strong activity in mechanical polishing and, therefore, microscratches may be generated on the polished surface of the substrate.

When a silica abrasive is used, the above-described problems are not caused and a polished surface having a small surface roughness R_a and less surface defects is obtained, but the silica abrasive is very low in the polishing rate and the processing efficiency is extremely poor.

In order to solve these problems, Japanese Unexamined Patent Publication (Kokai) No. 2002-150548 proposes to use an abrasive having a mechanochemical polishing activity on the amorphous phase moiety, such as ceria, in combination with an abrasive having a mechanical polishing activity mainly on the crystalline phase moiety, such as a silica abrasive. According to the polishing method of Japanese Unexamined Patent Publication (Kokai) No. 2002-150548, a glass substrate having high surface smoothness can be obtained at a high polishing rate.

In Japanese Unexamined Patent Publication (Kokai) No. 2002-150548, it is stated that if the content of the abrasive having a mechanical polishing activity on the

crystalline phase moiety of the crystallized glass substrate is less than 3 mass%, surface defects called pits (depression defect) are generated on the glass substrate and this is not preferred. In Japanese
5 Unexamined Patent Publication (Kokai) No. 2002-150548, it is also stated that if the content of the abrasive having a mechanochemical polishing activity on the amorphous phase moiety of the crystallized glass substrate is less than 1 mass%, the polishing rate disadvantageously
10 decreases.

The method of Japanese Unexamined Patent Publication (Kokai) No. 2002-150548 has some effects on the polishing of a glass substrate, particularly a crystallized glass substrate. Accordingly, the present invention provides a
15 polishing slurry useful for polishing a crystallized glass substrate at a lower cost. The present invention also provides a production method of a crystallized glass substrate with high surface smoothness for use in an information recording medium, and a production method of
20 a high-performance information recording medium.

DISCLOSURE OF THE INVENTION

The present inventors have found that, contrary to expectation, when a silica abrasive and a ceria abrasive, each in a relatively low concentration, are combined,
25 that is, when a silica abrasive of less than 3 mass% and a ceria abrasive of less than 1 mass% each based on the entire polishing slurry are combined, a polishing slurry having excellent effect with respect to defects and smoothness of the crystallized glass surface to be
30 polished can be obtained. The present invention has been accomplished based on this finding. The present invention is as follows.

(1) A polishing slurry comprising a silica abrasive and a ceria abrasive, wherein the silica abrasive content
35 is less than 3 mass% and the ceria abrasive content is less than 1 mass%, based on the entire polishing slurry.

(2) The polishing slurry as described in (1) above,

wherein the silica abrasive is a colloidal silica.

(3) The polishing slurry as described in (1) above, wherein the silica abrasive content is from more than 0.4 mass% to less than 2 mass% based on the entire polishing
5 slurry.

(4) The polishing slurry as described in (2) above, wherein the silica abrasive content is from more than 0.4 mass% to less than 2 mass% based on the entire polishing
slurry.

10 (5) A method for producing a crystallized glass substrate for an information recording medium, comprising providing a polishing slurry between a polishing member and a surface of a crystallized glass substrate having at least on the surface thereof an amorphous phase moiety
15 and a crystalline phase moiety, and effecting a relative motion between the glass substrate and the polishing member to polish the glass substrate, wherein the polishing slurry is the polishing slurry described in any one of (1) to (4) above.

20 (6) A crystallized glass substrate for an information recording medium, produced by the method described in (5) above.

(7) A method for producing an information recording medium, comprising forming a recording layer on a
25 crystallized glass substrate for an information recording medium, which is obtained by the method described in (5) above.

(8) The method for producing an information recording medium as described in (7) above, wherein the
30 recording layer is a magnetic layer.

BEST MODE FOR CARRYING OUT THE INVENTION

The polishing slurry of the present invention can be obtained by dispersing a ceria abrasive and a silica abrasive in a dispersion medium such as water.

35 The ceria abrasive usable for the polishing slurry of the present invention is generally available on the market and may have, for example, an average particle

diameter of 0.1 to 5 μm , particularly from 0.2 to 1.5 μm .

The silica abrasive usable for the polishing slurry of the present invention is generally available on the market as, for example, fumed silica, precipitated silica
5 or colloidal silica. Especially, colloidal silica may be used, and have, for example, an average particle diameter of 0.01 to 0.2 μm , particularly from 0.04 to 0.2 μm .

The dispersion medium usable for the polishing slurry of the present invention includes water and an
10 organic solvent, for example, a water-soluble organic solvent. The dispersion medium is preferably water.

The polishing slurry of the present invention may arbitrarily contain a surfactant as a dispersant. Examples of this surfactant include an anionic
15 surfactant, a cationic surfactant, a nonionic surfactant, an amphoteric ionic surfactant, and a combination thereof.

The polishing slurry of the present invention is useful particularly for the polishing of a crystallized
20 glass substrate having a crystalline phase moiety and an amorphous phase moiety, because the ceria abrasive in the polishing slurry of the present invention chemically and mechanically acts to exert a good polishing activity on the amorphous phase moiety of the crystallized glass
25 substrate and at the same time, the silica abrasive in the polishing slurry of the present invention mechanically acts to exert a good polishing activity even on the crystalline phase moiety where rapid polishing cannot be achieved by only the ceria abrasive.
30 Furthermore, the polishing slurry of the present invention can exert good polishing performance by using the abrasives in a low concentration and therefore, the cost of the polishing slurry can be decreased.

For polishing a glass substrate by using the
35 polishing slurry of the present invention, upper and lower platens each attached with a polishing cloth are used as the polishing member, a plurality of glass

substrates held by a carrier are interposed between these polishing members, and the upper and lower platens are rotated, whereby both surfaces of the glass substrate can be simultaneously polished. The polishing slurry of the present invention can also be used for other polishing methods using a brush, a polishing tape, a polishing pad or the like.

The polishing step may be performed in one stage or by dividing it into a plurality of stages. In the case of performing the polishing step by dividing it into a plurality of stages, a coarse polishing step of removing the processing deteriorated layer and flaws on the glass substrate surface, and a final polishing step of smoothing the glass substrate surface and removing surface defects are generally performed.

A polishing pad (hard polisher) comprising a relatively hard urethane foam or the like is used as the polishing member in the coarse polishing step, and a polishing pad (soft polisher) comprising a relatively soft artificial suede or the like is used as the polishing member in the final polishing step. The present invention is not limited by the polishing member used in combination with the polishing slurry of the present invention for the polishing of a glass substrate. For example, an urethane pad, a non-woven pad, an epoxy resin pad and the like can be used as the hard polisher, and a suede pad, a non-woven pad and the like can be used as the soft polisher.

The surface of the crystallized glass substrate obtained by the production method of a crystallized glass substrate for an information recording medium of the present invention has fewer defects and a small roughness and, therefore, when a recording layer is formed on the crystallized glass substrate, an information recording medium capable of high-density recording, such as a magnetic disc, a magneto-optical disc and an optical disc, can be obtained.

Particularly, in the case of producing a magnetic disc by using a magnetic layer as the recording layer, by virtue of fewer defects and a small roughness on the surface of the glass substrate, the spacing between the magnetic head and the magnetic disc can be made very small and in turn, the recording density can be increased. As for the magnetic layer used here, any magnetic material can be used, but a Co alloy is generally used. Incidentally, as is generally well known, a seed layer, an underlayer and an intermediate layer can be provided between the glass substrate and the magnetic layer, and a protective layer and a lubricant layer can be provided on the magnetic layer.

EXAMPLES

Example 1

A ceria abrasive-containing solution (SHOROX, produced by Showa Denko K.K., average particle diameter: 0.5 μm) having a solid content of 12 mass%, and a silica abrasive solution (Compol, produced by Fujimi Incorporated, average particle diameter: 0.08 μm) were added to water to form a polishing slurry having a ceria content of 0.6 mass% and a silica content of 0.2 mass%. A polishing test was performed by using the obtained polishing slurry.

A substrate for a magnetic recording medium was produced by using a crystallized glass obtained from a raw material of SiO_2 77%, Li_2O 11%, Al_2O_3 4% and MgO 3%.

In the production of this substrate for a magnetic recording medium, the raw material glass having the above-described composition was melted and mixed at a temperature of about 1,350 to 1,500°C by using a melting apparatus, and the melt was press-shaped and then cooled to obtain a disc-shaped sheet glass having a diameter of 66 mm ϕ and a thickness of 1 mm. This sheet glass was heat-treated at 540°C for about 5 hours to form crystal nuclei and then, crystal growth was allowed to proceed at

a temperature of 780°C for about 2 hours to obtain a crystallized glass. In this crystallized glass, the crystal phases were lithium disilicate and α -quartz, and particles resulting from aggregation of α -quartz were dispersed on the glass.

In the center of this sheet glass, a borehole was formed by using a cylindrical grindstone. Subsequently, the main surfaces of the substrate were subjected to a two-stage lapping process consisting of coarse lapping and precision lapping in a double-face polishing apparatus using diamond pellets, thereby adjusting the thickness and surface roughness of the substrate. Subsequently, the end face on the inner circumferential side facing the borehole of the substrate and the end face on the outer circumferential side were each chamfered with a grindstone by using an internal-external processing apparatus to form a chamfer.

The thus-processed glass substrate was processed for mirror polishing of respective end faces on the inner and outer circumferential sides. Subsequently, the main surfaces of the substrate were finally mirror-finished by using a double-face polishing apparatus (Model 16B, manufactured by SPEEDFAM Co., Ltd.). The polishing process was performed by two-stage polishing of coarse polishing and precision polishing.

In the coarse polishing, a cerium oxide powder-containing abrasive (ROX, produced by Showa Denko K.K.) was used as the abrasive, and a commercially available urethane pad was selected as the polishing pad. In the subsequent precision polishing, a cerium oxide powder-containing abrasive (ROX, produced by Showa Denko K.K.) and a colloidal silica-containing abrasive (Compol, produced by Fujimi Incorporated) were used as the abrasive, and a commercially available suede pad was selected as the polishing pad.

The obtained substrate was subjected to brush-scrub

cleaning and subsequently to immersion cleaning using an ultrasonic wave in combination to remove deposits on the surface, and then dried with IPA (isopropyl alcohol) vapor to obtain a glass substrate for a magnetic recording medium. The surface roughness and surface defect of the thus-obtained substrate were evaluated.

The polishing performance of the polishing slurry was evaluated by the following items (1) to (3). The results are shown in Table 1 below.

(1) Polishing Rate

The polishing rate was determined from the change in mass of the disc between before and after polishing. Incidentally, the polishing rate is shown by a ratio to a polishing rate obtained by a polishing slurry containing 0.6 mass% of ceria and not containing colloidal silica (Comparative Example 7) and taken as 100.

(2) Disc Surface Roughness: Ra

The surface roughness Ra (Å) was evaluated by using an atomic force microscope (D3000, manufactured by Digital Instruments) with a visual field of 10 μm.

(3) Surface Defects

The surface defects were evaluated by using ODT (Optical Defect Tester) RZ3500 manufactured by Hitachi Electronics Engineering Co., Ltd. In this measuring apparatus, laser light is made perpendicularly incident on the disc surface and from the phase difference of reflected light, the defects are detected. The measurement parameters used for this measurement were a disc size of 65 mm, a measuring radius of 11 to 31.5 mm and a slice of ±0.01 μm.

In this evaluation by ODT, a protruded defect is shown by P (positive) and a depressed defect is shown by N (negative). Accordingly, when P and N values are small, this means that the surface property of the disc is good.

Examples 2 to 5

A polishing test was performed in the same manner as

in Example 1 except that the contents of ceria and silica abrasives were changed to the contents shown in Table 1. The results are shown in Table 1.

Comparative Examples 1 to 7 and Reference Example

5 A polishing test was performed in the same manner as in Example 1 except that the contents of ceria and silica abrasives were changed to the contents shown in Table 1. The results are shown in Table 1.

Evaluation Results

10 As apparent from Table 1, in Examples 1 to 5 where the silica abrasive content was from more than 0 mass% to less than 3 mass% and the ceria abrasive content was from more than 0 mass% to less than 1 mass%, good polishing effects were obtained similarly to Reference Example
15 where the silica abrasive content and the ceria abrasive content both were high (corresponding to Japanese Unexamined Patent Publication (Kokai) No. 2002-150548).

 On the other hand, in Comparative Examples 1 to 5 where the ceria abrasive content was low (or the ceria
20 abrasive was not contained) and the silica abrasive content was high, the polishing rate was relatively low and the surface roughness Ra was large.

 In Comparative Example 7 where the silica abrasive was not contained, the measured values by ODT were large
25 and this reveals that many surface defects were present.

Table 1: Slurry Composition and Experimental Results

	Contents (mass%)		Polishing Rate	ODT Results		Ra
	Ceria	Colloidal Silica		P	N	
Ex. 1	0.6	0.2	96	12	11	3.3
Ex. 2	0.6	0.4	76	17	22	3.7
Ex. 3	0.6	0.8	56	19	18	3.6
Ex. 4	0.6	1.2	60	22	18	3.9
Ex. 5	0.6	2.0	76	7	16	4.3
Comp. Ex. 1	0.6	8.0	44	22	9	5.6
Comp. Ex. 2	0.4	8.0	40	7	14	4.4
Comp. Ex. 3	0.06	8.0	26	12	5	6.5
Comp. Ex. 4	0.01	8.0	29	5	2	8.0
Comp. Ex. 5	0.0	8.0	30	12	7	12.4
Comp. Ex. 6	9.4	1.6	82.5	20	15	3.6
Comp. Ex. 7	0.6	0.0	100	85	73	3.9
Ref. Ex	9.0	4.5	65.5	20	15	3.7

Thereafter, the obtained substrate was subjected to a texturing treatment with the use of a diamond slurry and a non-woven fabric and then mounted on a sputtering apparatus, and an under film consisting of a chromium alloy and a magnetic film consisting of a cobalt alloy were formed on both surfaces of the substrate by sputtering. Furthermore, a diamond-like carbon film was formed thereon by the CVD process and on this film, Fonblin Z-Tetraol (produced by Solvay Solexis) as a lubricant was coated to produce a magnetic recording medium. The total thickness of the films formed by sputtering was 90 nm, and the thickness of the film formed by CVD was 10 nm. In this way, 25 magnetic recording mediums were produced from the discs obtained in Examples 1 to 5 and the Reference Example.

The surface defect of these magnetic recording mediums was evaluated by using a Media Defect Evaluating Apparatus manufactured by Hitachi High-Technologies Corporation. The evaluation results revealed that the defect level of Examples 1 to 5 was similar to that of the Reference Example.

CLAIMS

1. A polishing slurry comprising a silica abrasive and a ceria abrasive, wherein the silica abrasive content is less than 3 mass% and the ceria abrasive content is less than 1 mass%, based on the entire polishing slurry.

2. The polishing slurry according to claim 1, wherein the silica abrasive is a colloidal silica.

3. The polishing slurry according to claim 1, wherein the silica abrasive content is from more than 0.4 mass% to less than 2 mass% based on the entire polishing slurry.

4. The polishing slurry according to claim 2, wherein the silica abrasive content is from more than 0.4 mass% to less than 2 mass% based on the entire polishing slurry.

5. A method for producing a crystallized glass substrate for an information recording medium, comprising providing a polishing slurry between a polishing member and a surface of a crystallized glass substrate having at least on the surface thereof an amorphous phase moiety and a crystalline phase moiety, and effecting a relative motion between the glass substrate and the polishing member to polish the glass substrate, wherein the polishing slurry is the polishing slurry according to any one of claims 1 to 4.

6. A crystallized glass substrate for an information recording medium, produced by the method according to claim 5.

7. A method for producing an information recording medium, comprising forming a recording layer on a crystallized glass substrate for an information recording medium, which is obtained by the method according to claim 5.

8. The method for producing an information recording medium according to claim 7, wherein the recording layer is a magnetic layer.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2005/016154

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl.⁷ C09K3/14, B24B37/00, C03C19/00, G11B5/73, 5/84

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl.⁷ C09K3/14, B24B37/00, C03C19/00, G11B5/73, 5/84

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
 Published unexamined utility model applications of Japan 1971-2005
 Registered utility model specifications of Japan 1996-2005
 Published registered utility model applications of Japan 1994-2005

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2003-31529 A (TOSHIBA Co., Ltd.) 2003.01.31, whole document, especially claims 1-3 & US 2003-17786 A1	1-5
Y	JP 2002-150548 A (HOYA Co., Ltd.) 2002.05.24, whole document, especially claims (Family:none)	1-8
Y	JP 2003-209076 A (HITACHI KASEI KOGYO Co., Ltd.) 2003.07.25, whole document, especially claim 1 and 【0006】 , 【0017】 , 【0018】 (Family:none)	1-8
Y	JP 2004-155914 A (YUSHIRO KAGAKU KOGYO Co., Ltd.) 2004.06.03, whole document, especially claims 1, 8 and 【0002】 , 【0010】 , 【0031】 , 【0032】 (Family:none)	1-8

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

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INTERNATIONALSEARCHREPORT

International application No.

PCT/JP2005/016154

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

The common subject-matter of claims 1-8 is considered the polishing slurry comprising a silica abrasive and a ceria abrasive, wherein the silica abrasive content is less than 3 mass% and the ceria abrasive content is less than 1 mass%, based on the entire polishing slurry. However, as such a polishing slurry is not novel (see [JP 2003-31529 A, claims 1-3]), it can not be a special technical feature. Therefore, there is no technical relationship among claims 1-8.

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
☐ No protest accompanied the payment of additional search fees.