

DESCRIPTION

[Title of Invention]

METHOD FOR REGENERATING USED POLISHING SLURRY, AND
5 METHOD FOR MANUFACTURING MAGNETIC-DISK GLASS SUBSTRATE

[Technical Field]

[0001] The present invention relates to a method for regenerating a used
polishing slurry and a method for manufacturing a magnetic-disk glass
10 substrate.

[Background Art]

[0002] In processing for polishing a glass substrate, sometimes, a cerium
polishing agent that mainly contains cerium oxide, for example, as polishing
abrasive particles is used. Because the cerium polishing agent is costly, it is
15 known that the used slurry is collected after the polishing processing and
regeneration processing for removing glass components such as glass sludge
(cullet) from the used polishing slurry is performed. In the processing for
regenerating the cerium polishing agent with solid-liquid separation by
centrifugation, the used polishing slurry is centrifuged, the solid portion
20 containing the polishing abrasive particles is collected, and liquid containing
glass components is discarded.

It is known that in the conventional processing for regenerating a used
polishing slurry, in order to easily collect the polishing abrasive particles,
magnesium salt is added to the used polishing slurry before solid-liquid
25 separation, or insonation is performed before or after solid-liquid separation (for
example, Patent Literatures 1 and 2).

[Citation List]

[Patent Literature]

[0004] [PLT1]: WO 2012/101868

30 [PLT2]: JP 2001-9723A

[Summary of Invention]

[Technical Problem]

[0004] When the processing for polishing a glass substrate was performed using the used polishing slurry regenerated by the conventional regeneration method, it was found that blemishes easily occur on main surfaces of the glass substrate. As a result of conducting studies regarding this point, it was found that blemishes occur on the glass substrate in the polishing processing because the polishing abrasive particles included in the used polishing slurry that has been regenerated include coarse particles or particles having a very hard grain boundary. Even if the particle size is not coarse, the polishing abrasive particles having a very hard grain boundary are not crushed up into small particles even if a load is received in the polishing processing, and as a result, there is a risk that blemishes will occur on the glass substrate.

An object of the present invention is to provide a method for regenerating a used polishing slurry according to which the solid portion generated by centrifuging a used polishing slurry can be sufficiently crushed, whereby it is possible to suppress the occurrence of blemishes on the glass substrate in the polishing processing. Also, an object of the present invention is to provide a method for manufacturing a magnetic-disk glass substrate according to which it is possible to suppress the occurrence of blemishes on the glass substrate in the polishing processing.

[Solution to Problem]

[0005] The present invention provides (1) to (21) below.

(1) An aspect of the present invention is a method for regenerating a used polishing slurry containing polishing abrasive particles used in processing for polishing a glass substrate, the method including:

a step of forming a mixture by adding a saccharide to the used polishing slurry;

a step of removing at least a portion of glass components included in the mixture by centrifuging the mixture; and

a step of crushing a solid portion obtained by the centrifugation.

[0006] (2) The method for regenerating a used polishing slurry according to (1) above, in which in the crushing step, the solid portion is crushed after water is added.

5 [0007] (3) The method for regenerating a used polishing slurry according to (1) or (2) above, in which the saccharide is selected from the group consisting of cellulose, pectin, protopectin, carrageenan, galactose, xanthan gum, locust bean gum, guar gum, psyllium seed gum, tamarind seed gum, tara gum, chitin, agarose, and a combination thereof.

10 [0008] (4) The method for regenerating a used polishing slurry according to any one of (1) to (3) above, in which the saccharide is insoluble in water.

[0009] (5) The method for regenerating a used polishing slurry according to any one of (1) to (4) above, in which an addition amount of the saccharide is 0.1 to 5 mass% with respect to a weight of the polishing abrasive particles.

15 [0010] (6) The method for regenerating a used polishing slurry according to any one of (1) to (5) above, in which a pH of the mixture is 5.5 to 13.

[0011] (7) The method for regenerating a used polishing slurry according to any one of (1) to (6) above, in which in the crushing step, ultrasound having a frequency of 16 to 120 kHz is emitted.

20 [0012] (8) The method for regenerating a used polishing slurry according to any one of (1) to (7) above, further including a step of drying the crushed solid portion.

[0013] (9) Another aspect of the present invention is a method for regenerating a used polishing slurry containing polishing abrasive particles used in processing for polishing a glass substrate, the method including:

25 a step of forming a mixture by adding a saccharide to the used polishing slurry;

a step of removing at least a portion of glass components included in the mixture by centrifuging the mixture; and

a step of crushing a solid portion obtained by the centrifugation,

30 in which the used polishing slurry is a used polishing slurry regenerated

with the method for regenerating a used polishing slurry according to any one of (1) to (8) above.

[0014] (10) Yet another aspect of the present invention is a method for regenerating a used polishing slurry containing polishing abrasive particles used in processing for polishing a glass substrate, the method including:

centrifugation processing for performing centrifugation using a treatment liquid containing the used polishing slurry,

in which the treatment liquid is adjusted to contain a massive object having a surface portion swollen by contact with water and an internal portion that is surrounded by the surface portion and that the water is blocked from entering.

[0015] (11) The method for regenerating a used polishing slurry according to (10) above, further including crushing processing for crushing a solid portion obtained by the centrifugation processing,

in which in the crushing processing step, the solid portion is crushed after water is added.

[0016] (12) The method for regenerating a used polishing slurry according to (11) above, in which in the crushing processing, ultrasound having a frequency of 16 to 120 kHz is emitted.

[0017] (13) The method for regenerating a used polishing slurry according to (11) or (12) above, further including drying processing for drying the crushed solid portion.

[0018] (14) The method for regenerating a used polishing slurry according to any one of (10) to (13) above, in which the treatment liquid is adjusted to contain the massive object using a saccharide selected from the group consisting of cellulose, pectin, protopectin, carrageenan, galactose, xanthan gum, locust bean gum, guar gum, psyllium seed gum, tamarind seed gum, tara gum, chitin, agarose, and a combination thereof.

[0019] (15) The method for regenerating a used polishing slurry according to (14) above, in which the saccharide is insoluble in water.

[0020] (16) The method for regenerating a used polishing slurry according to (14) or (15) above, in which an addition amount of the saccharide is 0.1 to 5 mass% with respect to a weight of the polishing abrasive particles.

5 [0021] (17) The method for regenerating a used polishing slurry according to any one of (10) to (16) above, in which a pH of the treatment liquid is 5.5 to 13.

[0022] (18) Still another aspect of the present invention is a method for regenerating a used polishing slurry containing polishing abrasive particles used in processing for polishing a glass substrate, the method including:

10 centrifugation processing for performing centrifugation using a treatment liquid containing the used polishing slurry,

 in which the treatment liquid is adjusted to contain a massive object having a surface portion swollen by contact with water and an internal portion that is surrounded by the surface portion and that the water is blocked from entering, and

15 the used polishing slurry is a used polishing slurry regenerated with the method for regenerating a used polishing slurry according to any one of (10) to (17) above.

20 [0023] (19) The method for regenerating a used polishing slurry according to any one of (1) to (18) above, in which the polishing abrasive particles contain cerium oxide or zirconium oxide as a main component.

[0024] (20) The method for regenerating a used polishing slurry according to any one of (1) to (19) above, in which the centrifugation is performed at 500 to 3000 G.

25 [0025] (21) Still another aspect of the present invention is a method for manufacturing a magnetic-disk glass substrate, the method including:

 polishing processing for polishing main surfaces of a glass plate using the polishing abrasive particles regenerated with the method for regenerating a used polishing slurry according to any one of (1) to (20) above.

[Advantageous Effects of the Invention]

30 [0026] According to the method for regenerating a used polishing slurry of the

present invention, the solid portion generated by centrifuging a used polishing slurry can be sufficiently crushed, whereby it is possible to suppress the occurrence of blemishes on a glass substrate in polishing processing. Also, according to the method for manufacturing a magnetic-disk glass substrate of the present invention, the occurrence of blemishes on a glass substrate in polishing processing is suppressed.

[Brief Description of Drawings]

[0027] FIG. 1(a) is a diagram schematically showing a state of water-containing aggregates and polishing abrasive particles in a used polishing slurry. FIG. 1(b) is a diagram schematically showing the solid portion generated by centrifugation.

FIG. 2 is a diagram schematically showing a state between polishing abrasive particles after water enters the water-containing aggregate.

[Description of Embodiments]

[0028] Hereinafter, a method for regenerating a used polishing slurry and a method for manufacturing a magnetic-disk glass substrate of the present invention will be described in detail.

[0029] Method for regenerating used polishing slurry

The regeneration method according to the present embodiment is a method for regenerating a used polishing slurry containing polishing abrasive particles (used polishing abrasive particles) used in processing for polishing a glass substrate, and includes a step of forming a mixture by adding a saccharide (for example, a water-insoluble polysaccharide that will be described later) to the used polishing slurry (mixture formation step), a step of removing at least a portion of glass components included in the mixture by centrifuging the mixture (removal step), and a step of crushing a solid portion obtained by the centrifugation (crushing step). The mixture formation step can be expressed as a step of making or preparing a mixture, or as a step of adjusting a treatment liquid that will be described later.

Although a case where a used polishing slurry used in polishing is

regenerated will be described as an example in the present embodiment, the regeneration method of the present embodiment can also be applied to a case where used polishing slurry (note that it contains abrasive particles) used in grinding is regenerated.

5 [0030] The polishing processing is performed by sandwiching the main surfaces of the substrate held by a carrier with a pair of polishing pads and moving the substrate and the polishing pads relative to each other by planet gear movement while supplying the polishing slurry between the polishing pads and the substrate, thereby polishing the main surfaces of the substrate. The processing
10 for polishing the glass substrate is performed using a double-side polishing apparatus that will be described later, for example. There is no particular limitation on the usage of a glass substrate that is to be subjected to the polishing processing, and the glass substrate is one type of glass substrate used as a panel for various display apparatuses such as LCD apparatuses, a mask
15 blank, and a magnetic disk for an HDD apparatus, for example.

The used polishing abrasive particles refer to polishing abrasive particles used in the processing for polishing a glass substrate at least once in the past. Examples of the polishing processing include first polishing and second polishing that are performed in a method for manufacturing a magnetic-
20 disk glass substrate, which will be described later, for example. Although there is no particular limitation on the polishing abrasive particles, a polishing agent that mainly contains cerium oxide (also referred to as cerium polishing agent, hereinafter) or a polishing agent that mainly contains zirconium oxide is used. The cerium polishing agent may contain oxide of another rare earth element
25 such as lanthanum, praseodymium, or neodymium as well as cerium. The polishing agent may further contain polishing agents other than a main polishing agent. The polishing abrasive particles have an average particle diameter D50 of 0.1 to 3 μm , for example, (D50 is a particle diameter at which a cumulative particle diameter measured by a laser diffraction/scattering particle
30 size distribution measurement method is equivalent to 50% accumulation

starting from the minute particle side).

The slurry is obtained by dispersing polishing abrasive particles in water, and may contain a dispersing agent or a water-insoluble polysaccharide that will be described later as well as the polishing abrasive particles, as appropriate.

5 Water used in the slurry may be water that contains impurities, such as tap water as well as pure water such as RO water, deionized water, or distilled water. Although there is no particular limitation on the dispersing agent, phosphoric acid-based dispersing agent is used, for example. Note that a slurry regenerated with the method for regenerating a used polishing slurry of the
10 present invention may be used as the used polishing slurry. In this case, the effect of reducing the cost of polishing abrasive particles can be obtained by repeatedly regenerating and using the used polishing slurry.

[0031] (a) Mixture formation step

In the mixture formation step, a mixture is formed by adding a
15 saccharide (for example, a water-insoluble polysaccharide) to the used polishing slurry. It is preferable that the saccharide has the property of swelling due to contact with water. A massive object (a so-called lump) is easily formed in water due to this property. Also, the saccharide preferably has a low solubility in neutral water (also simply referred to as water hereinafter) at room
20 temperature (20°C), and it is more preferably insoluble therein. The lump can be stably present for a long period due to the saccharide having such a property. From the above-described viewpoint, among saccharides, cellulose, chitin; galactose, locust bean gum, tara gum; protopectin, carrageenan, xanthan gum, guar gum, psyllium seed gum, tamarind seed gum, tara gum, chitin, agarose are
25 preferable, and cellulose, chitin; galactose, locust bean gum, and tara gum are more preferable, and cellulose and chitin are most preferable. Saccharides can be used in combination. Note that if cellulose is used, it is preferable to use crystalline cellulose.

[0032] Here, a water-insoluble polysaccharide will be described as an example
30 of a water-insoluble saccharide. Note that if "water-insoluble" or "insoluble" is

used in this specification, it encompasses "partially dissolving in water" as well as "not dissolving in water at all". Specifically, "partially dissolving in water" refers to the amount of the saccharide dissolving in water being 30 wt% or less, or 20 wt% or less, or 10 wt% or less of the addition amount. Therefore, "water-insoluble polysaccharide" or "insoluble polysaccharide" described later can be expressed as polysaccharide that dissolves in water in an amount that is part or less of the addition amount (30 wt%, 20 wt%, or 10 wt%). Water-insoluble polysaccharide having the property of not dissolving in water but absorbing water and swelling, and being water-absorbable and water retentive so as to be present as an aggregate containing water (also referred to as a water-containing aggregate or massive object) in the used polishing slurry is preferably used. Examples of such a substance include cellulose, pectin, protopectin, carrageenan xanthan gum, locust bean gum, guar gum, psyllium seed gum, tamarind seed gum, tara gum, chitin, agarose, and a combination thereof. Among these, cellulose, pectin, protopectin, and carrageenan are preferably used. Also, in order that a lump is easily formed in water and is stably present for a long period, cellulose, chitin; locust bean gum, tara gum; protopectin, carrageenan, xanthan gum, guar gum, psyllium seed gum, tamarind seed gum, tara gum, chitin, and agarose are preferable, cellulose, chitin; galactose, locust bean gum, and tara gum are more preferable, and cellulose and chitin are most preferable. Note that in the description below, a water-insoluble polysaccharide (that is, a polysaccharide that dissolves in water in an amount that is part or less of the addition amount) will be described as an exemplary saccharide.

Addition of such a water-insoluble polysaccharide to the used polishing slurry and the mixture containing water-containing aggregates makes it easy to crush the solid portion generated by centrifugation performed later. Note that the water-insoluble polysaccharide has the functional effect of preventing the solid portion (also referred to as deposition) generated by centrifugation from hardening excessively.

[0033] The addition amount of the insoluble polysaccharide is preferably 0.1 to

5 mass% with respect to the weight of the polishing abrasive particles (dry weight). If the addition amount is less than 0.1 mass%, there are cases where it takes time to crush the solid portion obtained after the centrifugation. Also, if the insoluble polysaccharide is added in an amount of more than 5 mass%, the insoluble polysaccharide is likely to enter between the polishing abrasive particles and a substrate to be polished, and sometimes, the polishing rate decreases after regeneration.

The type and addition amount of the insoluble polysaccharide is appropriately determined to achieve a state in which the insoluble polysaccharide present in the solid portion generated by centrifugation can further absorb moisture, or in other words, a state in which the outer portion of a water-containing aggregate retains water and the inner portion thereof is dry (a so-called lump state), for example. "Dry state" herein refers to a state in which the water-containing aggregate can further absorb water or a state in which it has not sufficiently absorbed water after centrifugation. Therefore, when the water-containing aggregate in the lump state is crushed by applying an external force thereto, a dried portion appears. In the description below, a water-containing aggregate refers to, unless otherwise stated, an aggregate whose outer portion retains water and whose inner portion is dry, or in other words, a massive object having a surface portion (outer portion) that swells due to contact with water and an internal portion (inner portion) that is surrounded by the surface portion and that the water is blocked from entering.

[0034] It is preferable to add the insoluble polysaccharide to the used polishing slurry in a state in which it is prepared as a water-containing aggregate in advance. The water-containing aggregates are obtained by adding an insoluble polysaccharide to water and subjecting the mixture to appropriate stirring and insonation. At this time, the inner portion of the water-containing aggregate is desirably in a state in which it has not sufficiently absorbed water. Although there is no particular limitation on the size of the water-containing aggregate, the maximum length is 1 to 300 μm , for example. It is preferable that the

water-containing aggregate has a particle diameter larger than the particle diameter of polishing abrasive particles.

The state shown in FIG. 1(a) of the used polishing slurry can be prepared by adding an insoluble polysaccharide and then appropriately stirring the mixture so that polishing abrasive particles adhere to the surfaces of the water-containing aggregates. FIG. 1(a) is a diagram schematically showing a state of the water-containing aggregates and polishing abrasive particles in the used polishing slurry. In FIG. 1(a), multiple polishing abrasive particles 3 are attached to the surfaces of water-containing aggregates 1. Note that the sizes of the water-containing aggregates 1 and the particles 3 have been adjusted in FIG. 1 in order to give a simple description.

Note that the used polishing slurry containing the water-containing aggregates (massive objects) is, in other words, a treatment liquid that is subjected to centrifugation in the centrifugation processing of a removal step, which will be described later. A step of forming polishing abrasive particles or a mixture can be expressed as adjusting the treatment liquid used in the centrifugation processing so as to contain the massive objects.

[0035] The pH of the mixture of the insoluble polysaccharide and the used polishing slurry that is obtained by adding the insoluble polysaccharide to the used polishing slurry is preferably adjusted to 5.5 or more, more preferably 7 or more, and even more preferably 10 or more. If the pH is less than 5.5, there are cases where the effect of removing the glass components that are present in the slurry and the glass components attached to the polishing abrasive particles will decrease and the polishing rate of performing the processing for polishing a glass substrate using the regenerated used polishing slurry cannot be sufficiently recovered. On the other hand, under strong alkaline conditions where the pH exceeds 13, there are cases where alkaline earth metal ions of the glass components (for example, Mg^{2+} , Ca^{2+}) and aluminum ions (Al^{3+}) will form hydroxides, be deposited, and be mixed into the centrifuged solid portion, resulting in a decrease in the polishing rate or the occurrence of blemishes on

the glass substrate. Therefore, the pH is preferably 13 or less. Although the pH is adjusted using a strong base such as sodium hydroxide or potassium hydroxide, and a strong acid such as sulfuric acid or phosphoric acid, as appropriate, it is not particularly limited to the method using these.

5 [0036] Note that it is preferable that coarse particles, which are foreign matter, are removed by filtering from the used polishing slurry before the mixture formation step. The coarse particles are solid components that are mixed into the used polishing slurry by the polishing processing, such as scraps of polishing pads, polishing abrasive particles that are dried and coarsened, glass pieces like
10 glass chips, for example. Removing such foreign matter makes it possible to prevent solid components other than the polishing abrasive particles from mixing into the solid portion generated by the "removal step" that will be performed later. The coarse particles herein refer to particles having a maximum length (diameter) of 15 μm or more.

15 [0037] (b) Removal step

In the removal step, at least a portion of the glass components included in the mixture is removed by centrifuging the above-described mixture (treatment liquid) (also referred to as centrifugation processing). Centrifugation is performed using a centrifugal separator. Centrifugation is
20 preferably performed at 500 to 3000 G. Accordingly, the solid portion and the glass sludge component can be effectively separated. Also, centrifugation is more preferably performed under conditions of 600 to 1800 G. The radius of rotation and the rotation rate of the centrifugal separator are selected so as to meet the range of such a centrifugal force. Also, the centrifugation time is
25 appropriately adjusted in accordance with the centrifugal force of the centrifugation, and is 15 minutes to 1 hour, for example. Note that conditions such as the centrifugal force of centrifugation, the centrifugation processing time, dehydration processing time, and the scraping time are appropriately determined with consideration given to the slurry concentration and the slurry
30 amount for performing the centrifugation processing, the removal amount of

glass components, the weight or collection ratio of the solid portion, and the like. In order to prevent the removal amount of glass components or the property of the solid portion being crushed from deteriorating, the moisture amount of the solid portion is preferably 10 to 40 mass%. The moisture amount is appropriately adjusted with consideration given to the slurry concentration and the slurry amount for performing the centrifugation processing, the removal amount of glass components, the weight or the collection ratio of the solid portion, and the like. Centrifugation may be performed while the separated liquid is cyclically supplied, that is, the separated liquid is returned to the centrifugal separator.

[0038] In the removal step, the mixture is separated into the solid portion containing polishing abrasive particles and the liquid (is subjected to solid-liquid separation). Also, the solid portion may be collected by centrifuging again the liquid that has undergone the solid-liquid separation until the concentration of the polishing abrasive particles reaches a given value or less. If centrifugation is performed again, an insoluble polysaccharide may be added. Note that the liquid will ultimately be discarded. Note that glass components (two types, namely, dissolved components and minute particles) derived from the glass sludge (cullet) are present in the liquid, and thus the glass components (two types, namely, dissolved components and minute particles) are removed in the removal step.

[0039] Here, the functional effect of the insoluble polysaccharide included in the separated solid portion will be described.

As shown in FIG. 1(b), the solid portion is deposited such that the polishing abrasive particles 3 overlap each other with insoluble polysaccharides (not shown) interposed therebetween, as described later. FIG. 1(b) is a diagram schematically showing the solid portion generated by centrifugation. Note that in order to give a simple description, the interval between the polishing abrasive particles 3 is omitted in FIG. 1(b). Also, the sizes of polishing abrasive particles are shown as being uniform for the convenience of

description. The polishing abrasive particles attached to the surfaces of water-containing aggregates present in the used polishing slurry in the mixture formation step enter the inner portions of the water-containing aggregates due to the functional effect of the centrifugal force by centrifugation, and accordingly, the polishing abrasive particles 3 come close to each other, and as shown in FIG. 2, they are deposited with insoluble polysaccharides 5 interposed between the particles 3. FIG. 2 is a diagram schematically showing the state of the polishing abrasive particles and the insoluble polysaccharides included in the solid portion, and is a diagram focusing on a portion of the solid portion. Note that for a simple description, the interval between the polishing abrasive particles 3 in FIG. 1 and the interval between the particles 3 shown in FIG. 2 have been adjusted. Also, for a simple description, the sizes of the particles 3 and the insoluble polysaccharides 5 have been adjusted in FIG. 2.

FIG. 2 shows a case where cellulose is used as the insoluble polysaccharide. As shown in FIG. 2, the polishing abrasive particles 3 are deposited in a state in which the particles 3 are in contact with fibrous cellulose but not in contact with each other. The water-containing aggregates gradually become smaller than the above-described lump state in the centrifugation process, play a role as a cushion when the interval between the polishing abrasive particles 3 decreases, and have the functional effect of suppressing the contact between the polishing abrasive particles 3. Even if centrifugation is performed, the solid portion that is easily crushed later due to such functional effects of the insoluble polysaccharide is produced.

[0040] (c) Crushing step

In the crushing step, the solid portion obtained by the centrifugation is crushed (also referred to as crushing processing). Although the crushing of the solid portion is suitably performed by generating ultrasound cavitation using a homogenizer, for example, it may be performed using an ultrasound tank. If the homogenizer is used, it is preferable to generate ultrasound having a frequency of 16 kHz to 120 kHz. Crushing time with ultrasound having a

frequency in this range is 1 to 60 minutes, for example. Accordingly, the particle size distribution of the polishing abrasive particles can be made suitable for re-use.

[0041] The crushing of the solid portion is preferably performed after water is added to the solid portion. Because the water-containing aggregates have room to further absorb water, water enters the dried inner portion due to the addition of water, and accordingly, the solid portion can be easily crushed, resulting in a better particle size distribution of the polishing abrasive particles. Although there is no particular limitation on the addition amount of water, the addition amount is such that the weight of the solid portion is 1 to 40 wt%, for example. Water may be added by adding a substance having moisture.

[0042] The above-described removal steps and the above-described crushing steps may be performed alternately once or twice, for example.

[0043] The removal of foreign matter may be performed once or several times after the crushing step. Filtering may be used as the method for removing foreign matter, for example. The foreign matter removed here is polishing abrasive particles that have not been sufficiently crushed, for example. The size of foreign matter to be removed is 15 μm or more, for example. Also, if foreign matter is removed several times, the particle diameter of the polishing abrasive particles may be adjusted by crushing or the like before the next removal of foreign matter.

[0044] In order to increase the handling property of the polishing abrasive particles, the used polishing slurry that was regenerated may be dried (drying step or drying processing). Drying is performed by spray drying, for example.

If the used polishing slurry is left to stand, there is a risk that the polishing abrasive particles in the used polishing slurry will precipitate to the bottom of the container and aggregate, producing coarse particles. The occurrence of blemishes caused by the coarse particles can be suppressed by drying in order to prevent such a phenomenon.

[0045] According to the regeneration method of the present embodiment, the

addition of the insoluble polysaccharide to the used polishing slurry that was collected makes it possible to prevent the solid portion from being hardened by centrifugation and to obtain the solid portion that can be easily crushed.

It is known that in order to prevent the polishing abrasive particles
5 included in the slurry from forming a hard cake, insoluble polysaccharides are added to a slurry before processing for polishing a glass substrate. However, because the insoluble polysaccharides added before the polishing processing are sufficiently dispersed in the slurry by loads received from polishing pads, a carrier, the glass substrate, and the like in the polishing processing, and their
10 internal portions are hydrated, and thus the insoluble polysaccharides are not in the dry state. With the regeneration method of the present embodiment, the solid portion is easily crushed by adding an insoluble polysaccharide to the used polishing slurry before centrifugation such that the insoluble polysaccharide (water-containing aggregate) whose internal portion is in the dry state is formed,
15 preferably allowing the dried portion to absorb moisture in the crushing step, and applying shearing stress thereto. In this manner, with the regeneration method of the present embodiment, the solid portion is sufficiently prevented from being hardened by adding an insoluble polysaccharide again after the used polishing slurry is collected. Also, according to the regeneration method of the
20 present embodiment, because the solid portion can be easily crushed, whereby it is possible to inhibit polishing abrasive particles having a coarse particle size and polishing abrasive particles whose grain boundary is very hard from remaining in the slurry. Thus, if polishing processing is performed using the used polishing slurry that was regenerated, the occurrence of blemishes on main
25 surfaces of the glass substrate can be suppressed.

Note that the method for regenerating a used polishing slurry of the present embodiment can be expressed as a method for manufacturing a slurry in that at least a particle diameter of polishing abrasive particles is adjusted. Also, in a case where a glass substrate that is to be subjected to polishing
30 processing is a glass substrate used as a magnetic disk and polishing abrasive

particles include cerium oxide (ceria), the regeneration method of the present embodiment can be expressed as a method for regenerating a ceria slurry for a magnetic-disk glass substrate.

[0046] Method for manufacturing magnetic-disk glass substrate

5 Next, a method for manufacturing a magnetic-disk glass substrate according to the present embodiment will be described.

 This manufacturing method includes polishing processing for polishing main surfaces of a glass plate using polishing abrasive particles regenerated by the above-described method for regenerating a used polishing slurry.

10 Although a case where the used polishing slurry regenerated with the method for regenerating a used polishing slurry is used for polishing will be described as an example in the present embodiment, this used polishing slurry can also be used for grinding.

[0047] To give a description of the outline of the manufacturing method of the present embodiment, first, molding processing for forming a plate-shaped glass blank having a pair of main surfaces is performed. The glass blank serves as a raw material for a magnetic-disk glass substrate. Next, rough-grinding processing is performed on this glass blank. Thereafter, shape processing is performed on the glass blank to form a glass substrate, and edge surface polishing processing is performed thereon. Then, precision grinding processing is performed on the glass substrate using fixed abrasive particles. Thereafter, first polishing processing and second polishing processing are performed on the glass substrate. Note that although the above-described processes are performed in the above-described flow in the present embodiment, the present embodiment is not limited to the above flow or type of processes, and these processes may be omitted as appropriate. Hereinafter, each of the processes will be described.

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[0048] (a) Processing for molding glass blank

 In molding processing, molding is performed with a press molding method, for example. A disk-shaped glass blank can be obtained by a press

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molding method. The glass blank may be manufactured using a known molding method such as a downdraw method, a redraw method, or a fusion method, instead of the pressing method. A disk-shaped glass substrate, which is the base of a magnetic-disk glass substrate, can be obtained by appropriately performing shape processing, which will be described later, on the plate-shaped glass blank produced with these methods.

[0049] (b) Rough grinding processing

Next, rough grinding processing is performed. In the rough grinding processing, main surfaces on both sides of the glass blank are ground while the glass blank is held in a carrier (not shown) of a double-sided grinding apparatus. Specifically, the glass blank, which is held in the holding hole of the carrier, is held between the upper surface plate and the lower surface plate, and the glass substrate and the surface plates are moved relative to each other by moving one or both of the upper surface plate and the lower surface plate while a grinding liquid containing a grinding agent is supplied, and thereby both main surfaces of the glass substrate are ground. Loose abrasive particles are used as a grinding agent, for example. In rough grinding processing, grinding is performed such that the glass blank is brought approximately closer to a target substrate thickness and a target flatness of the main surfaces. Note that although the rough grinding processing is performed in accordance with the dimensional accuracy or the surface roughness of the molded glass blank, it can be omitted as appropriate.

[0050] (c) Shape processing

Next, shape processing is performed. In the shape processing, a circular hole is formed in the glass blank using a known processing method to obtain a disk-shaped glass substrate having the circular hole. Thereafter, chamfering is performed on edge surfaces of the glass substrate. Chamfering is performed on the edge surfaces on the inner circumferential side and the outer circumferential side of the glass substrate. Chamfering forms side wall surfaces orthogonal to the main surfaces and chamfered surfaces (interposing

surfaces) that connect the side wall surfaces and the main surfaces on the edge surfaces of the glass substrate.

[0051] (d) Edge surface polishing processing

Next, processing for polishing edge surfaces of the glass substrate is performed. In the edge surface polishing processing, polishing is performed by supplying a polishing liquid containing loose abrasive particles between a polishing brush and the edge surfaces of the glass substrate and moving the polishing brush and the glass substrate relative to each other in the thickness direction of the glass substrate. The inner circumferential side edge surface and the outer circumferential side edge surface of the glass substrate are polished and formed into mirror surfaces by edge surface polishing processing.

[0052] (e) Precision grinding processing

Next, precision grinding processing is performed on the main surfaces of the glass substrate. In the precision grinding processing, grinding is preferably performed on the main surfaces of the glass substrate using a double-sided grinding apparatus in which fixed abrasive particles are stuck to the surface plates. Specifically, both main surfaces of the glass substrate are ground similarly to the above-described rough grinding processing, except that grinding is performed using fixed abrasive particles instead of the above-described loose abrasive particles. The glass substrate is held by the carrier of the above-described embodiment and is ground while undergoing a planet gear movement in the double-sided grinding apparatus. Although the main surfaces of the glass substrate are ground by bringing grinding surfaces of the surface plates to which fixed abrasive particles are stuck into contact with the main surfaces of the glass substrate, grinding may be performed using loose abrasive particles, instead of this.

[0053] (f) First polishing processing

Next, first polishing processing is performed on the main surfaces of the glass substrate. In the first polishing processing, the main surfaces on both sides of the glass substrate are polished using a known double-sided polishing

apparatus while the glass substrate is held by the carrier. A known apparatus can be used as a double-sided polishing apparatus for performing the first polishing processing. The apparatus includes a pair of upper and lower surface plates, an internal gear that is sandwiched between the upper and lower surface plates, a sun gear provided on the lower surface plate, and a plurality of carriers that engage with the internal gear and the sun gear. Polishing pads are stuck to the surface plates. The glass substrate is sandwiched between the upper and lower surface plates in a state of being held in the holding hole of the carrier, and the upper and lower surface plates are rotated, and thereby the carrier that holds the glass substrate revolves while rotating, as a result of which this apparatus performs a planet gear movement. Accordingly, the glass substrate and the polishing pads are moved relative to each other, and the main surfaces of the glass substrate are polished. In particular, in the first polishing processing, polishing is performed by bringing the polishing pads stuck to the surface plates into contact with the main surfaces of the glass substrate, using loose abrasive particles. During the first polishing processing, the slurry containing the loose abrasive particles is supplied between the glass substrate and the polishing pads via a pipe (not shown) from a supplying tank (not shown) of the double-side polishing apparatus. After being supplied between the glass substrate and the polishing pads, the slurry may be collected and again cyclically supplied between the glass substrate and the polishing pads during polishing processing on the same or a different batch. The used polishing slurry regenerated by the above-described regeneration method is used as the slurry containing the loose abrasive particles. Although there is no particular limitation on the loose abrasive particles, cerium oxide abrasive particles (the above-described cerium polishing agent), zirconia abrasive particles, a combination thereof, or the like are used, for example.

[0054] The first polishing processing removes cracks and warping remaining on the main surfaces or removes minute surface unevenness (roughness or waviness) occurring on the main surfaces in the case where grinding is

performed as pretreatment with fixed abrasive particles, for example. Appropriately adjusting the machining allowance amount makes it possible to reduce the surface roughness of the main surfaces, for example, an arithmetic average roughness Ra, while preventing the shape of the edge portions of the main surfaces from excessively recessing or protruding. Also, the polishing abrasive particles that are used in the first polishing processing and are included in the used polishing slurry have good particle size distribution due to the above-described regeneration method, and therefore the occurrence of blemishes on the main surfaces of the glass substrate is suppressed.

[0055] Note that the slurry collected in the first polishing processing may be further regenerated by the above-described regeneration method, or the regenerated collected slurry (used polishing slurry) may be further used in the processing for polishing another glass substrate.

[0056] (g) Second polishing (mirror-polishing) processing

Next, second polishing processing is performed. The second polishing processing is for performing mirror-polishing on the main surfaces. Although in the second polishing processing, a double-sided polishing apparatus and a polishing method that are similar to those used in the first polishing processing may be used, it is preferable that the size of the polishing abrasive particles is smaller than that of the polishing abrasive particles used in the first polishing processing. Doing so makes it possible to reduce the roughness of the main surfaces while preventing the shape of end portions of the main surfaces from excessively recessing or protruding. After the second polishing processing, the glass substrate is removed from the double-side polishing apparatus and cleaned.

After the second polishing processing is performed and then the glass substrate is cleaned (final cleaning), an inspection step of inspecting the quality of the glass substrate may be performed.

[0057] Working Examples

In order to confirm the effects of the present invention, the following

experiment was performed. Specifically, used polishing slurries were regenerated in accordance with procedures described below by variously changing the conditions of used polishing slurries used in centrifugation as shown in Tables 1 to 3 (Samples 1 to 15), removal amounts of glass components in the used polishing slurries that were regenerated and the polishing rates were measured, and the degrees of the occurrence of blemishes occurring when glass substrates were polished using the regenerated slurries (regeneration slurries) were evaluated.

[0058] The used polishing slurries were regenerated by performing steps 1 to 7 below in the stated order. A slurry collected after the first polishing processing of the above-described embodiment was performed once using a slurry containing unused cerium polishing agent was used as the used polishing slurry. The first polishing processing was performed under the same polishing conditions as those of the first polishing processing, which will be described later, using a double-side polishing apparatus. Note that processes other than the first polishing processing were carried out with the content of the above-described embodiment.

[0059] Step 1: foreign matter removal step

Foreign matter such as glass chips was removed by filtering. The size of foreign matter to be removed was set to 15 μm or more.

[0060] Step 2: solid-liquid separation step

After saccharides were added to the used polishing slurries in accordance with Tables 1 to 3 and the pH was adjusted, solid-liquid separation was performed using a centrifugal separator, liquid was discarded, and the solid portions were collected. Centrifugation was performed at 1000 G for 60 minutes.

Note that the type and addition amounts of the saccharides are shown in alignment in the field "Type and addition amount of saccharide to be added" in Tables 1 to 3. Also, the addition amounts of the saccharides are expressed in mass ratio with respect to the weight (dry weight) of the unused polishing

abrasive particles.

[0061] Step 3: Crushing step

The solid portions were crushed for 30 minutes by adding water such that the concentrations of the solid portions reached 10 to 30 wt% and
5 generating ultrasound cavitation at a frequency of 20 kHz with a homogenizer.

Step 4: foreign matter removal step

Foreign matter was removed by filtering from the slurries obtained by step 3. The size of foreign matter to be removed was set to 15 μm or more.

Step 5: particle diameter adjustment step

10 The particle diameters of the cerium polishing agents included in the slurries obtained in step 4 were adjusted using a wet cyclone as a classifier. Note that this step is performed to remove abrasive particles that hardly contribute to polishing due to having an extremely small diameter. Therefore, not carrying out this step has no great influence. If this step is not carried out,
15 the polishing rate just slightly decreases.

Step 6: foreign matter removal step

Foreign matter was removed by filtering from the slurries obtained in step 5. The size of foreign matter to be removed was set to 15 μm or more.

Step 7: drying step

20 The slurries were subjected to spray drying.

[0062] Evaluation of removal amount of glass components

The content of glass components included in the cerium polishing agent in each sample obtained by step 7 was analyzed by X-ray fluorescence analysis. The content of glass components was evaluated as a ratio of the weight of SiO_2 with respect to the weight of the cerium polishing agent (SiO_2 /cerium polishing agent ratio). The SiO_2 /cerium polishing agent ratio is a parameter indicating the removal amount of glass components, and indicates the weight of SiO_2
25 attached to the cerium polishing agent with respect to the weight of the cerium polishing agent included in the used polishing slurry that was regenerated.
30 The smaller the value of the SiO_2 /polishing abrasive particle ratio is, the greater

the effect of removing glass components is. The weight ratio of SiO_2 with respect to the cerium polishing agent is shown in Table 2.

[0063] Evaluation of polishing rate and degree of occurrence of blemishes

The first polishing processing of the above-described embodiment was performed on the glass substrates under the polishing conditions described below, using a slurry (used polishing slurry) containing cerium polishing agent of each sample and using an apparatus 400 used in the first polishing processing described in JP 2012-133882A as a double-side polishing apparatus provided with a planetary gear mechanism. Note that suede polishing pads made of polyurethane foam were stuck to the surfaces of the upper and lower surface plates.

- Concentration of polishing abrasive particles: 10 wt%
- Load on glass substrate by upper surface plate and lower surface plate: 100 g/cm²
- Polishing time: 60 minutes

[0064] Amorphous aluminosilicate glass having a nominal size of 2.5 inches and a composition described below was used as the glass substrate. That is, glass containing, as main components, SiO_2 in an amount of 58 wt% or more and 68 wt% or less, Al_2O_3 in an amount of 5 wt% or more and 20 wt% or less, Li_2O in an amount of 0 wt% or more and 10 wt% or less, Na_2O in an amount of 4 wt% or more and 12 wt% or less, K_2O in an amount of 0 wt% or more and 6 wt% or less, and ZrO_2 in an amount of 1 wt% or more and 10 wt% or less was used.

[0065] After the above-described first polishing processing was performed and the glass plates were cleaned and dried, the polishing rates were calculated based on differences in weights of glass substrates before and after polishing. Also, the light of a condensing lamp was applied to the cleaned and dried glass substrates in a dark room, and the number of scratches and pits on main surfaces (both surfaces) of the glass substrates were visually counted. If the degree of blemish occurrence was less than 20/surface, such a glass substrate was evaluated as level A, if it was 20 or more and less than 100/surface, such a

glass substrate was evaluated as level B, and if it was 100 or more/surface, such a glass substrate was evaluated as level C. If the degree of blemish occurrence is level A or B, the occurrence of blemishes can be suppressed and such a glass substrate has no problems in terms of practical use. The polishing rate was indicated with a relative value using Sample 2 as 100%. If the relative value is 90% or more, such a glass substrate is preferable in terms of practical use. The results are shown in Tables 1 and 3.

[0066] [Table 1]

Sample No.	Conditions of used polishing slurry subjected to centrifugation		Degree (level) of blemish occurrence
	pH of slurry	type and addition amount (wt%) of saccharide to be added	
1	10	none	C
2		cellulose, 0.5	A
3		cellulose, 2.0	
4		pectin, 0.5	
5		protopectin, 0.5	
6		carrageenan, 0.5	
7	7	none	C
8		cellulose, 0.5	A
9		pectin, 0.5	
10	3	none	C
11		cellulose, 0.5	B
12		pectin, 0.5	

[0067] [Table 2]

Sample No.	Conditions of used polishing slurry subjected to centrifugation		SiO ₂ /cerium polishing agent ratio (wt%)
	pH of slurry	type and addition amount (wt%) of saccharide to be added	
2	10	cellulose, 0.5	0.4
8	7	cellulose, 0.5	0.6
11	3	cellulose, 0.5	1.5
13	5.5	cellulose, 0.5	0.8

[0068] [Table 3]

Sample No.	Conditions of used polishing slurry subjected to centrifugation		Polishing rate
	pH of slurry	type and addition amount (wt%) of saccharide to be added	
2	10	cellulose, 0.5	100
3	10	cellulose, 2.0	100
14	10	cellulose, 5.0	90
15	10	cellulose, 7.0	85

5 [0069] As shown in Table 1, it was found that if saccharides were added to the used polishing slurries in centrifugation, the occurrence of blemishes on the glass substrate was suppressed. In particular, it was found that compared to the case where the pH was adjusted to less than 5.5 (Samples 11 and 12), if the used polishing slurries were produced by adjusting the pH to 5.5 or more in the mixture formation step (Samples 2 to 6, 8, and 9), the occurrence of blemishes was more suppressed. It is conceivable that this is because glass components that hinder polishing were sufficiently removed from the surfaces of the polishing abrasive particles by adjusting the pH to 5.5 or more, resulting in the original surfaces of the polishing abrasive particles.

15 As shown in Table 2, it was found that if a used polishing slurry was produced by adjusting the pH to 5.5 or more in centrifugation in the mixture formation step, the glass components were more sufficiently removed from the

polishing abrasive particles of the used polishing slurry that was regenerated. In this case, a higher polishing rate can be obtained.

As shown in Table 3, it was found that if the addition amount of the saccharide is 5.0 mass% or less, the polishing rate is high.

5 [0070] In addition to the above-described experiment, a regeneration slurry was obtained (Sample 16) similarly to Sample 2 except that crushing processing was performed using a vibration dryer without adding water in step 3. With regard to this slurry, when the degree of blemish occurrence was evaluated with a method similar to the above, the glass substrate was evaluated as level B. The
10 other characteristics were similar to those of Sample 2.

Also, a regeneration slurry was obtained (Sample 17) similarly to Sample 2 except that step 5 was not performed. With regard to this slurry, when the polishing rate was calculated with a method similar to the above, the relative value thereof was 96 when the polishing rate of Sample 2 was set to 100.
15 The other characteristics were similar to those of Sample 2.

[0071] Evaluation of yield

Regeneration slurries were produced by variously changing sugars to be added to the used polishing slurries as shown in Table 4 (Samples 18 to 30), and performing steps 1 to 7 above in the stated order, and the first polishing
20 processing was performed on the glass substrates having sizes and compositions similar to the above, using the produced regeneration slurries, and processes other than the first polishing processing were carried out up to final cleaning with the same procedures as the above-described embodiment. 1000 glass substrates were produced for each sample with this method. The yields of the
25 produced glass substrates were evaluated using a laser surface defect inspection apparatus. Regarding each sample, the addition amount of the saccharide was 0.5 wt% and the pH of the used polishing slurry used when centrifugation was performed was adjusted to 7. The yields are shown in Table 4.

In this evaluation, substrates having surfaces with blemishes that were
30 formed in the first polishing processing and not removed in the second polishing

processing and are not visible can be detected and excluded, and a difference in the degree of the occurrence of blemishes on samples can be expressed relatively clearly. That is, it is conceivable that the number of relatively deep blemishes of the blemishes formed in the first polishing processing is reflected in the yield.

5 [0072] [Table 4]

Sample No.	Added sugar	Yield (%)
18	cellulose	98
19	chitin	97
20	locust bean gum	93
21	tara gum	92
22	galactose	91
23	pectin	87
24	protopectin	84
25	carrageenan	85
26	xanthan gum	83
27	guar gum	86
28	psyllium seed gum	85
29	tamarind seed gum	84
30	agarose	83

[0073] As described above, although a method for regenerating a used polishing slurry and a method for manufacturing a magnetic-disk glass substrate according to the present invention have been described in detail, the present invention is not limited to the above-described embodiment, and it will be appreciated that various improvements and modifications can be made without departing from the gist of the present invention.

[Descriptions of Reference Numerals]

[0074] 1 Water-containing aggregate (massive object)
 15 3 Polishing abrasive particles

5 Insoluble polysaccharide (polysaccharide that dissolves in an amount that is part or less of the addition amount)

CLAIMS

1. A method for regenerating a used polishing slurry containing polishing abrasive particles used in processing for polishing a glass substrate, the method
5 comprising:
 - a step of forming a mixture by adding a saccharide to the used polishing slurry;
 - a step of removing at least a portion of glass components included in the mixture by centrifuging the mixture; and
 - 10 a step of crushing a solid portion obtained by the centrifugation.
2. The method for regenerating a used polishing slurry according to claim 1, wherein
 - 15 in the crushing step, the solid portion is crushed after water is added.
3. The method for regenerating a used polishing slurry according to claim 1 or 2, wherein
 - the saccharide is selected from the group consisting of cellulose, pectin, protopectin, carrageenan, galactose, xanthan gum, locust bean gum, guar gum,
 - 20 psyllium seed gum, tamarind seed gum, tara gum, chitin, agarose, and a combination thereof.
4. The method for regenerating a used polishing slurry according to any one of claims 1 to 3, wherein
 - 25 the saccharide is insoluble in water.
5. The method for regenerating a used polishing slurry according to any one of claims 1 to 4, wherein
 - an addition amount of the saccharide is 0.1 to 5 mass% with respect to a
 - 30 weight of the polishing abrasive particles.

6. The method for regenerating a used polishing slurry according to any one of claims 1 to 5, wherein

a pH of the mixture is 5.5 to 13.

5

7. A method for regenerating a used polishing slurry containing polishing abrasive particles used in processing for polishing a glass substrate, the method comprising:

10 a step of forming a mixture by adding a saccharide to the used polishing slurry;

a step of removing at least a portion of glass components included in the mixture by centrifuging the mixture; and

a step of crushing a solid portion obtained by the centrifugation,

15 wherein the used polishing slurry is a used polishing slurry regenerated with the method for regenerating a used polishing slurry according to any one of claims 1 to 6.

8. A method for regenerating a used polishing slurry containing polishing abrasive particles used in processing for polishing a glass substrate, the method
20 comprising:

centrifugation processing for performing centrifugation using a treatment liquid containing the used polishing slurry,

25 wherein the treatment liquid is adjusted to contain a massive object having a surface portion swollen by contact with water and an internal portion that is surrounded by the surface portion and that the water is blocked from entering.

9. The method for regenerating a used polishing slurry according to any one of claims 1 to 8, wherein

30 the polishing abrasive particles contain cerium oxide or zirconium oxide

as a main component.

10. A method for manufacturing a magnetic-disk glass substrate, comprising

- 5 polishing processing for polishing main surfaces of a glass plate using the polishing abrasive particles regenerated with the method for regenerating a used polishing slurry according to any one of claims 1 to 9.