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(74) Agents: **AOKI, Atsushi** et al.; A. Aoki, Ishida & Associates, Toranomon 37 Mori Bldg., 5-1, Toranomon 3-chome, Minato-ku, Tokyo 1058423 (JP).

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(71) Applicants (*for all designated States except US*): **SHOWA DENKO K.K.** [JP/JP]; 13-9, Shibadaimon 1-chome, Minato-ku, Tokyo 1058518 (JP). **YAMAGUCHI SEIKEN KOGYO K.K.** [JP/JP]; 153 Aza Horogo, Narumicho, Midori-ku, Nagoya-shi, Aichi 4580801 (JP).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **MIYATA, Norihiko** [JP/JP]; c/o Shiojiri Plant Showa Denko K.K., 1, Oaza Soga, Shiojiri-shi, Nagano 3996461 (JP). **ANDO, Junichiro** [JP/JP]; c/o Yamaguchi Seiken Kogyo K.K., 153 Aza Horogo, Narumicho, Midori-ku, Nagoya-shi, Aichi 4580801 (JP). **HONG, Gong-Hong** [KR/JP]; c/o Yamaguchi Seiken Kogyo K.K., 153 Aza Horogo, Narumicho, Midori-ku, Nagoya-shi, Aichi 4580801 (JP).

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(54) Title: POLISHING COMPOSITION AND POLISHING METHOD

(57) Abstract: A polishing composition for polishing a magnetic disk substrate, characterized by comprising water, abrasive grains, a polishing accelerator, and a surface cleaning agent which is a compound comprising a main chain of 2 to 6 carbon atoms and optionally an oxygen atom, said compound having a plurality of hydroxyl groups. The composition is capable of providing a magnetic disk substrate having low surface roughness, generating no protrusion or scratch, preventing deposition of residues on the substrate surface, realizing high-density recording, and being capable of polishing the substrate at a cost-effective rate.

WO 2004/104122 A2

DESCRIPTION

POLISHING COMPOSITION AND POLISHING METHOD

5 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. §119(e)(1), of the filing date of the Provisional Application No.60/475,807 filed on June 5, 2003, pursuant
10 to 35 U.S.C. §111(b).

TECHNICAL FIELD

The present invention relates to a polishing composition for use in precise polish-finishing of metal,
15 plastic, glass, or similar material and, more particularly, to a polishing composition suitable for polishing a magnetic disk substrate or a similar substrate for use in a memory device of a computer or a similar device. Still more particularly, the present
20 invention relates to a polishing composition for a magnetic disk substrate or a similar substrate, which composition can provide a polished magnetic disk surface with high precision suitable for use in combination with a magnetic head that flies at a low height.

25 BACKGROUND ART

Among external storage devices for a computer or a word processor, a magnetic disk (a memory hard disk) is widely used as a high-speed accessible means. One typical example of the magnetic disk is produced by
30 subjecting an Al alloy substrate to electroless plating of NiP to form a substrate, polishing a surface of the substrate, and sputtering onto the substrate a Cr alloy undercoat layer, a Co alloy magnetic layer, and a carbon protective layer, in the order given.

35 However, if protrusions having a height that exceeds the flying height of the magnetic head remain on the magnetic disk surface, the magnetic head traveling at

high speed collides with the protrusions while flying at a predetermined height. As a result, such protrusions cause damage to the magnetic head. In addition, if the magnetic disk substrate has protrusions, scratches, etc.,
5 when a Cr alloy undercoat layer, a Co alloy magnetic layer, or a similar layer is formed on the substrate, protrusions are replicated on the film and flaws attributed to the scratches result, whereby the magnetic disk fails to be provided with a smooth surface having
10 high precision. Thus, the substrate must be subjected to precision polishing in order to enhance the precision of the disk surface.

One possible reason for the generation of the aforementioned protrusions is that abrasive grains and
15 chemical agents are deposited and, after polishing, remain on the NiP surface whose surface activity has been elevated by the agents serving as a polishing accelerator. Particularly when submicron particles and nano-size particles have been once deposited on the
20 surface, these particles are difficult to remove only through application of generally employed external force. Thus, a surface cleaning agent is an effective material required for preventing deposition of such particles.

In connection with the polishing of the magnetic
25 disk substrate, there have been proposed many polishing compositions that can remove protrusions or buff the protrusions down to as low a height as possible without making the substrate prone to scratches.

Among documents disclosing such polishing
30 compositions, Japanese Patent Application Laid-Open (*kokai*) No. 9-204657 discloses use of a composition including colloidal silica with aluminum nitrate and an anti-gelling agent, and Japanese Patent Application Laid-Open (*kokai*) No. 9-204658 discloses use of a composition
35 including fumed silica and aluminum nitrate. These compositions disclosed in the above documents employ finely-divided particles of silicon oxide with low

hardness as the abrasive grains so that the surface precision can be easily obtained. However, achieving a higher polishing rate suitable for real production is difficult.

5 Japanese Patent Application Laid-Open (*kokai*) No. 10-204416 discloses, as means for accelerating polishing rate, use of various kinds of oxidizing agents as well as use of an Fe salt. However, the attained polishing rate is still insufficient, to achieve a higher polishing rate
10 in actual production.

Japanese Patent Application Laid-Open (*kokai*) No. 2001-89749 discloses that polyoxyethylene alkyl ether phosphates and polyoxyethylene aryl ether phosphates serve as agents for reducing residues remaining on the
15 surface. However, the polymeric compounds are expensive, and a washing effect is not so high.

In order for a composition for polishing an aluminum magnetic disk substrate to realize high-density magnetic recording, the composition is required to provide a high
20 precision disk surface that enables a head to fly at a low height.

Thus, an object of the present invention is to provide a composition for polishing a magnetic disk substrate, the composition being capable of providing a
25 magnetic disk substrate having low surface roughness, generating no protrusion or scratches, preventing deposition of residues on the substrate surface, realizing high-density recording, and being capable of polishing the substrate at a cost-effective rate.

30 SUMMARY OF THE INVENTION

The gist of the present invention resides in a polishing composition which can attain the above object and which contains water, abrasive grains, a polishing accelerator, and a surface cleaning agent of a compound
35 comprising a main chain having 2 to 6 carbon atoms and optionally an oxygen atom, said compound having a plurality of hydroxyl groups. Accordingly, the present

invention provides the following.

(1) A polishing composition characterized by comprising water, abrasive grains, a polishing accelerator, and a surface cleaning agent which is a compound comprising a main chain having 2 to 6 carbon atoms and optionally an oxygen atom, said compound having a plurality of hydroxyl groups.

(2) The polishing composition according to (1) above, wherein the surface cleaning agent is at least one species selected from the group consisting of glycerin, ethylene glycol, diethylene glycol, triethylene glycol, and propylene glycol.

(3) The polishing composition according to (1) or (2) above, wherein the composition contains the surface cleaning agent in an amount of 0.1 to 15 mass% based on the entirety of the polishing composition.

(4) The polishing composition according to any one of (1) to (3) above, wherein the abrasive grains are at least one species selected from the group consisting of alumina, titania, silica, and zirconia.

(5) The polishing composition according to any one of (1) to (4) above, wherein the abrasive grains have a mean particle size falling within a range of 0.001 to 0.5 μm .

(6) The polishing composition according to any one of (1) to (5) above, wherein the abrasive grains are colloid particles.

(7) The polishing composition according to any one of (1) to (6) above, wherein the polishing accelerator is at least one species selected from the group consisting of a phosphorus-containing inorganic acid or a salt thereof; a not-phosphorus-containing inorganic acid, or a salt of the second inorganic acid; an organic phosphonic acid chelate compound; and an oxidizing agent.

(8) The polishing composition according to any one of (1) to (7) above, wherein the polishing composition contains, as an polishing accelerator, (i) a phosphorus-

containing inorganic acid or a salt thereof; (ii) a not-phosphorus-containing inorganic acid, or a salt of the not-phosphorus-containing inorganic acid and/or an organic phosphonic acid chelate compound; and (iii) an oxidizing agent.

(9) The polishing composition according to (7) or (8) above, wherein the phosphorus-containing inorganic acid is phosphoric acid or phosphonic acid.

(10) The polishing composition according to any one of (7) to (9) above, wherein the not-phosphorus-containing inorganic acid is at least one species selected from the group consisting of nitric acid, sulfuric acid, amidosulfuric acid, and boric acid.

(11) The polishing composition according to any one of (7) to (10) above, wherein the oxidizing agent is a peroxide or a nitrate salt.

(12) The polishing composition according to (1) above, wherein the peroxide is at least one species selected from the group consisting of hydrogen peroxide, a perborate salt, and a persulfate salt.

(13) The polishing composition according to any one of (1) to (12) above, which has a pH falling within a range of 1 to 5.

(14) The polishing composition according to any one of (7) to (13) above, wherein the amount of abrasive grains falls within a range of 3 to 30 mass%.

(15) The polishing composition according to any one of (7) to (14) above, wherein the amount of inorganic acid or a salt thereof falls within a range of 0.1 to 8 mass%.

(16) The polishing composition according to any one of (7) to (15) above, wherein the amount of organic phosphonic acid chelate compound falls within a range of 0.01 to 10 mass%.

(17) The polishing composition according to any one of (7) to (16) above, wherein the amount of oxidizing agent falls within a range of 0.2 to 5 mass%.

(18) The polishing composition according to any one of (7) to (13) above, wherein the amount of abrasive grains falls within a range of 3 to 30 mass%, the amount of inorganic acid or a salt thereof falls within a range of 0.1 to 8 mass%, the amount of organic phosphonic acid chelate compound falls within a range of 0.01 to 10 mass%, and the amount of oxidizing agent falls within a range of 0.2 to 5 mass%.

(19) The polishing composition according to any one of (1) to (18) above, further containing a surfactant.

(20) The polishing composition according to any one of (1) to (19) above, further containing an anticorrosive.

(21) The polishing composition according to any one of (1) to (20) above, further containing a preservative.

(22) The polishing composition according to any one of (1) to (21) above, further containing an anti-gelling agent.

(23) A kit of two compositions for forming the polishing composition as set forth in any one of (1) to (22) above by mixing or by mixing and diluting the two compositions.

(24) The kit according to (23) above, in which one of said two compositions comprises abrasive grains and water, and the other one comprises an inorganic acid, an oxidizing agent and a surface cleaning agent.

(25) The kit according to (23) or (24) above, for transportation or storage.

(26) A composition which forms the polishing composition according to any one of (1) to (22) above by dilution.

(27) A method of employing the composition as set forth in any one of (23) to (26) above as a composition for transportation and storage.

(28) A method of polishing a magnetic disk substrate, the method employing a polishing composition as recited in any one of (1) to (26) above.

(29) A method for preparing a polishing composition by mixing the two compositions of the kit as set forth in any one of (23) to (25) above.

5 (30) A method for polishing characterizing by mixing the two compositions of the kit as set forth in any one of (23) to (25) above and employing the obtained composition for polishing.

10 (31) A method for polishing characterizing by preparing a composition having a higher concentration than that for polishing, diluting said prepared composition to obtain the polishing composition as set forth in any one of claims (1) to (22) above, and employing the obtained composition for polishing.

15 (32) A method of producing a magnetic disk substrate, the method employing the method as recited in any one of (28), (30) and (31) above.

(33) The method according to (32) above, in which the magnetic disc substrate has a recording density of 3 Gbit/inch² or more.

20 (34) The method according to (32) or (33) above, in which the magnetic disc substrate is an aluminum substrate.

25 (35) The method according to (32) or (33) above, in which the magnetic disc substrate is a NiP-plated aluminum substrate.

MODES OF CARRYING OUT THE INVENTION

The surface cleaning agent employed in the present invention is a compound (hydrocarbons) comprising a main chain of 2 to 6 carbon atoms and optionally an oxygen
30 atom, said compound having a plurality of hydroxyl groups. The effect of the cleaning agent includes preventing deposition of abrasive grain residues and polishing agent residues on a magnetic disk substrate and washing out of the residues from the disk substrate.
35 Once deposited on the substrate, such residues cannot be removed through post-washing. Although the mechanism of the effect has not been completely elucidated, the effect

is similar to that of alcohol or a like compound. Namely, a plurality of hydroxyl groups of the cleaning agent are believed to enhance wettability of the substrate surface, and thus promote the effect of preventing deposition of and removal of the residues, thereby playing a key role in providing a high-quality polished surface. An alcoholic agent having a simple hydroxyl structure provides weak wettability and tends to evaporate or diffuse, whereas a polymeric agent having many carbon atoms assumes the form of solid agent, because of poor solubility in water. Therefore, the aforementioned agent having a plurality of hydroxyl groups and an appropriate molecular weight is thought to be a suitable cleaning agent.

Specific examples of preferred cleaning agents include glycerin, ethylene glycol (EG: abbreviation in Table 2), diethylene glycol (DEG: abbreviation in Table 2), triethylene glycol, and propylene glycol.

The amount of the surface cleaning agent based on the entire polishing composition is preferably 0.1 to 15 mass% (unless otherwise specified, in the present specification "%" hereinafter refers to "mass%"), more preferably 0.5 to 10%, most preferably 1 to 5%.

No particular limitation is imposed on the type of abrasive grains contained as an abrasive in the polishing composition of the present invention, and alumina, titania, silica, zirconia, or a similar compound can be employed. Also, no particular limitation is imposed on the crystal form of the abrasive grains. For example, there can be preferably employed any of alumina (i.e., aluminum oxide) having a crystalline form of α , γ , δ , η , θ , κ , or χ ; titania (i.e., titanium oxide) having a crystalline form of rutile, anatase, or brookite; silica (i.e., silicon oxide) including colloidal silica, fumed silica, and white carbon; and zirconia (i.e., zirconium oxide) having a monoclinic system, a tetragonal system,

or an amorphous form. These abrasive grains in the form of colloid particles are more preferred, as the effect of preventing micro-scratches is enhanced.

5 The abrasive grains generally have a mean particle size of 0.001 to 0.5 μm , preferably 0.01 to 0.2 μm , more preferably 0.02 to 0.2 μm , most preferably 0.03 to 0.2 μm . In addition, as mentioned above, abrasive grains in the form of colloidal particles are more preferable. In the present invention, a value measured by means of a
10 laser Doppler frequency analysis particle size distribution analyzer (Microtrac UPA150, product of by Honeywell, Inc.) is used as the mean particle size of abrasive grains.

With increase in particle size of the abrasive
15 grains, gelation and aggregation of fine particles can be prevented more easily, while the probability of coarse particles existing increases, thereby generating scratches in the course of polishing. Decrease in particle size readily causes the above-mentioned gelation
20 and aggregation, which also generates scratches during polishing.

When the abrasive grains are contained in the polishing composition in an amount of less than 3%, increase in the polishing rate is difficult. With
25 increase in concentration of the abrasive grains, the polishing rate increases. However, when the concentration of the abrasive grains exceeds 30%, increase in the polishing rate becomes less, and in addition, gelation of the abrasive grains occurs readily,
30 particularly when the abrasive grains are in the form of colloidal particles. In view of economical efficiency, the concentration of 30% or less is preferable for practical use. Thus, the concentration of the abrasive grains in the polishing composition preferably falls
35 within a range of 3 to 30%, more preferably 5 to 15%.

The polishing composition preferably contains, as an

polishing accelerator, a phosphorus-containing inorganic acid or a salt thereof; a not-phosphorus-containing inorganic acid not containing phosphorus, or a salt of the not-phosphorus-containing inorganic acid; an organic phosphoric acid-based chelate compound; and/or an oxidizing agent.

Notably, the aforementioned "not-phosphorus-containing inorganic acid, or a salt thereof" (may be referred to as "another inorganic acid or a salt thereof") contained in the polishing composition of the present invention may be substituted by or may be used in combination with the "organic phosphonic acid chelate compound" described below.

In the polishing composition of the present invention, the phosphorus-containing inorganic acid or a salt thereof may be used in combination with the not-phosphorus-containing inorganic acid or a salt of the not-phosphorus-containing inorganic acid. The phosphorus-containing inorganic acid contains a phosphorus atom as an element composing the compound and is preferably phosphoric acid or phosphonic acid. The phosphorus-containing inorganic acid includes a derivative thereof. The phosphorus-containing inorganic acid may be used singly or in combination of two or more species.

Examples of the not-phosphorus-containing inorganic acid which can be used in combination with the phosphorus-containing inorganic acid include nitric acid, hydrochloric acid, sulfuric acid, chromic acid, carbonic acid, amidosulfuric acid, and boric acid. Of these, nitric acid, sulfuric acid, amidosulfuric acid, and boric acid are preferred. These acids include derivatives thereof.

Examples of the salt of the phosphorus-containing inorganic acid and the salt of the not-phosphorus-containing inorganic acid include those acid salts containing a metal such as Li, Be, Na, Mg, K, Ca, Ti, V,

Cr, Mn, Fe, Co, Ni, Cu, Zn, Al, Zr, Nb, Mo, Pd, Ag, Hf, Ta, or W. These salts can be produced by, for example, dissolving an oxide or carbonate of the aforementioned metal in the phosphorus-containing inorganic acid or the not-phosphorus-containing inorganic acid.

The total amount of the phosphorus-containing inorganic acid or a salt thereof and the not-phosphorus-containing inorganic acid or a salt of the not-phosphorus-containing inorganic acid contained in the polishing composition of the present invention is 0.1 to 8%, preferably 0.2 to 6%, more preferably 0.4 to 4%. When the total amount of inorganic acids and salts thereof is less than 0.1%, the effect of preventing micro-scratches and increasing the polishing rate is small, whereas when the amount exceeds 8%, the pH may drop excessively, thereby requiring acid resistance in a polishing machine. The ratio in amount of the phosphorus-containing inorganic acid or a salt thereof to the not-phosphorus-containing inorganic acid or a salt of the not-phosphorus-containing inorganic acid is preferably 1 mol : 0.1 to 5 mol. When the total amount of the not-phosphorus-containing inorganic acid and a salt of the not-phosphorus-containing inorganic acid is less than 0.1 mol%, dispersibility of a solid in the slurry may decrease, thereby increasing micro-scratches, whereas when the total amount is in excess of 5 mol, the pH may decrease excessively, thereby inflicting considerable damage on a polishing machine.

The polishing composition of the present invention contains at least one specific inorganic acid or a salt thereof. When the composition contains a phosphorus-containing inorganic acid or a salt thereof serving as a primary essential component, the polishing rate and the effect of preventing micro-scratches increase.

Although the function and effect of the at least one specific inorganic acid or a salt thereof used in the present invention have not been elucidated, one

conceivable effect is provision of a good dispersion state of the polishing composition.

As one species of the aforementioned polishing accelerator, the organic phosphonic acid chelate compound described, for example, in Japanese Patent Application
5 Laid-Open (*kokai*) No. 2001-131535, can be employed.

Specifically, the organic phosphonic acid chelate compound of the present invention may be diethylenetriaminepentamethylenephosphonic acid,
10 phosphonobutanetricarboxylic acid (abbreviated as PBTC in Table 2), phosphonohydroxyacetic acid, hydroxyethyldimethylphosphonic acid, aminotrismethylenephosphonic acid (abbreviated as NTMP in Table 2), hydroxyethanediphosphonic acid (abbreviated as
15 HEDP in Table 2), ethylenediaminetetramethylenephosphonic acid, hexamethylenediaminetetramethylenephosphonic acid, and salts thereof.

The amount of the organic phosphonic acid chelate compound preferably falls within a range of 0.01 to 10%
20 based on the entirety of the polishing composition. When the amount is too small, the effect of enhancing polishing rate is less, whereas when the amount is excessively large, surface defects such as pits and protrusions may be generated and, if the compound is in
25 the form of colloid particles, gelling readily occurs. More preferably, the amount falls within a range of 0.05 to 5%.

Although the organic phosphonic acid chelate compound may be used singly, the compound may also be
30 used in combination with another polishing accelerator so as to enhance the polishing rate.

The oxidizing agent which can be incorporated in the polishing composition of the present invention is preferably a peroxide or a nitrate salt. Typically, as
35 the peroxide, preferred are hydrogen peroxide perborate salt (e.g., sodium perborate), persulfate salt (e.g., ammonium persulfate), and nitrate salt (e.g., ammonium

nitrate). The effects of the oxidizing agent include increase in polishing rate and reduction in surface roughness. Although the effect of these oxidizing agents has not been completely elucidated, one conceivable effect is that the oxidizing agent serves as an etchant with respect to the Ni-P surface.

Meanwhile, in the case where the aforementioned not-phosphorus-containing inorganic acid or a salt thereof has an oxidizing action, the acid or the salt may also be used as the oxidizing agent. For example, a nitrate salt is used to serve as both the not-phosphorus-containing inorganic acid salt and the oxidizing agent. Needless to say, even when another inorganic acid or a salt thereof has an oxidizing action, an oxidizing agent differing from the inorganic acid or a salt thereof may be incorporated in the composition.

The oxidizing agent (e.g., hydrogen peroxide) is incorporated in the composition in an amount of 0.2 to 5%, preferably 0.5 to 2%. When the amount of the oxidizing agent is less than 0.2%, the effects of increasing polishing rate and reducing surface roughness are insufficient, whereas when the amount is in excess of 5%, these effects are not further enhanced.

When the not-phosphorus-containing inorganic acid or a salt thereof also is used as an oxidizing agent, the total amount of the not-phosphorus-containing inorganic acid or a salt thereof is equal to the sum of the preferable total amount (0.1 - 8%) of the not-phosphorus-containing inorganic acid or a salt thereof and the preferable amount (0.2 - 5%) of the oxidizing agent. Accordingly, the total amount of the phosphorus-containing inorganic acid or a salt thereof and the not-phosphorus-containing inorganic acid or a salt thereof in this case is preferably 0.3 to 13%, more preferably 0.7 to 8%.

When the aforementioned composition contains, in addition to the not-phosphorus-containing inorganic acid

or a salt thereof also serving as an oxidizing agent, an oxidizing agent (not an inorganic acid or salt thereof) and an inorganic acid not acting as an oxidizing agent or a salt thereof, the amount of the inorganic acid acting also as an oxidizing agent or a salt thereof is determined and then the upper limit of the amount of the oxidizing agent (not an inorganic acid or a salt) can be determined. For example, when the composition contains phosphoric acid, a nitrate salt (acting as a salt of the not-phosphorus-containing inorganic acid and also as an oxidizing agent), and hydrogen peroxide in combination, the amount of the nitrate salt is determined to that of the not-phosphorus-containing inorganic acid salt, and the upper limit amount of hydrogen peroxide, as the oxidizing agent, is determined to be 5%. In another case, when another inorganic acid or a salt thereof not acting as an oxidizing agent is used in combination with a nitrate salt, the upper limit amount of the nitrate salt is determined to be 5%, and then the total amount of the inorganic acid not acting as the oxidizing agent or a salt thereof can be determined. In each case, the total amount of the phosphorus-containing inorganic acid or a salt thereof, the not-phosphorus-containing inorganic acid or a salt of the not-phosphorus-containing inorganic acid, and the oxidizing agent is preferably 0.3 to 13%, more preferably 0.7 to 8%.

The polishing composition of the present invention preferably has a pH falling within a range of 1 to 5, more preferably 1 to 4, most preferably 1 to 3. By controlling the liquid of the composition to a more acidic state, oxidation of Ni is accelerated, thereby increasing the polishing rate. However, as an excessively low pH value causes a problem of corrosion of an apparatus or other problems, the pH is preferably 1 to 3, more preferably 1 to 2.

In addition to the aforementioned components, other components such as a surfactant, an anticorrosive, and a

preservative may be incorporated into the polishing composition of the present invention. However, the type and amount of each optional component should be carefully selected so as to avoid causing gelation.

5 In order to prevent gelation, an anti-gelling agent may be incorporated into the polishing composition of the present invention. As the anti-gelling agent, at least one species selected from among a phosphonic acid compound, phenanthroline, and aluminum acetylacetonate is
10 preferably used. Specific examples of the phosphonic acid compound include 1-hydroxyethane-1,1-diphosphonic acid ($C_2H_6O_7P_2$), and aminotrimethylenephosphonic acid ($C_2H_{12}O_9P_3N$). Examples of phenanthroline include 1,10-phenanthroline monohydrate ($C_{12}H_8N_2 \cdot H_2O$). Examples of the
15 aluminum acetylacetonate include acetylacetonatoaluminum ($AlCH(COCH_3)_3$). These compounds are preferably incorporated in a total amount of 2% or less.

Notably, the aforementioned concentrations of components are suitable concentrations upon polishing a
20 magnetic disk substrate. Thus, in an effective way, the polishing composition is prepared at component concentrations higher than the aforementioned concentrations for transportation, storage or other steps, and upon use, is diluted so that the
25 concentrations fall within the aforementioned ranges.

Similar to conventional polishing compositions, the polishing composition of the present invention can be prepared by suspending polishing materials in water and adding, to the suspension, inorganic acids such as
30 phosphoric acid and nitric acid, an oxidizing agent, a surface cleaning agent, etc. Upon use, the composition containing all components may be diluted. For example, in order to enhance the stability during transportation or storage, it is preferred that the polishing
35 composition is prepared as a bid of two compositions, depending on ingredients, for example, a slurry and a solution, more specifically one composition comprising

water and abrasive grains, the other composition comprising water, an inorganic acid, an oxidizing agent and a surface cleaning agent, which are mixed together just prior to use, if necessary after transportation and storage.

The polishing composition of the present invention can be suitably applied to a substrate; for example, a substrate for high recording density (generally, recording density of ≥ 3 Gbit/inch²) such as a magnetic recording disk for a magnetic head employing magnetoresistance (MR) effect. Also, the composition is effectively applied to a magnetic disk of lower recording density, from the viewpoint of enhancement of reliability.

No particular limitation is imposed on the magnetic hard disk substrate to which the polishing composition is applied. However, application of the composition of the present invention to an aluminum (including aluminum alloy) substrate, particularly an aluminum substrate which is plated with NiP through, for example, electroless plating, is advantageous, as a high-quality polished surface can be obtained in an industrial process.

For carrying out the polishing method, a polishing pad generally employed in polishing by use of a slurry-like polishing material is caused to press against a magnetic disk substrate, and either the pad or the substrate is rotated in a sliding manner while a slurry is fed to a gap between the pad and the substrate.

The magnetic disk produced from a substrate which has been polished by use of the polishing composition of the present invention has remarkably excellent smoothness; i.e., very few micro-defects such as micro-pits and micro-scratches, and a surface roughness (Ra) of 3 Å or less.

EXAMPLES

The present invention will next be described in

detail by way of examples, which should not be construed as limiting the invention thereto. Any modification to the examples performed without deviating the spirit of the invention described above and below falls within the technical scope of the present invention.

The type and characteristics of the polishing materials employed in Examples and Comparative Examples are shown in Table 1.

<Examples 1 to 5>

Water; inorganic acid 1 (phosphorus-containing inorganic acid or a salt thereof, hereinafter the same expression will be adopted), inorganic acid 2 (not-phosphorus-containing inorganic acid or a salt thereof, hereinafter the same expression will be adopted), and/or an organic phosphonic acid chelate compound; an oxidizing agent, and a surface cleaning agent were added to colloidal silica (Syton HT-50, product of Du Pont Kabushiki Kaisha) in the proportions shown in Table 2, to thereby prepare a variety of aqueous polishing compositions. Polishing was performed under polishing conditions using a polishing machine as described below. The results are shown in Table 2.

<Examples 6 and 7>

Each of white carbon (E-150J, product of Nippon Silica Industrial Co., Ltd.) and fumed silica (AEROSIL-50, product of Nippon Aerosil Co., Ltd.) was ground in a media agitating mill, and coarse particles were removed through classification, whereby the corresponding silicon oxide particles having a mean particle size of 0.1 μm were produced. Subsequently, water, inorganic acid 1, inorganic acid 2, an oxidizing agent, and a surface cleaning agent were added to the respective silicon oxide particles in the respective proportions shown in Table 2, to thereby prepare a variety of aqueous polishing compositions. Polishing was performed under polishing conditions using a polishing machine as described below. The results are shown in Table 2.

<Examples 8 to 10>

Each of titanium oxide (Supertitania F-4, product of Showa Titanium Co., Ltd.), alumina, and zirconia was ground in a media agitating mill, and coarse particles were removed through classification, whereby the corresponding oxide (titanium oxide, alumina, and zirconia) particles having a mean particle size of 0.2 μm were produced. Subsequently, water, inorganic acid 1, inorganic acid 2 or an organic phosphonic acid chelate compound, an oxidizing agent, and a surface cleaning agent were added to the respective oxide particles in the respective proportions shown in Table 2, to thereby prepare a variety of aqueous polishing compositions. Polishing was performed under the polishing conditions, and using a polishing machine, as described below. The results are shown in Table 2.

The mean particle size was determined by means of a laser Doppler frequency analysis particle size distribution analyzer (Microtrac UPA150, product of by Honeywell, Inc.). The measured values of particle size are shown in Table 1. The pH values of the compositions were measured by means of a hydrogen ion concentration meter with glass electrodes (D-13, Product of by HORIBA, Ltd.).

<Polishing conditions>

As a substrate, a 3.5-inch aluminum disk plated with NiP through electroless plating was employed.

<Polishing machine and polishing conditions>

Polishing machine: 4-way double-sided polishing machine

Polishing pad: Suede type (Polytex DG, product of Rodel, Inc.)

Number of revolution of lower surface plate: 60 rpm

Slurry feeding speed: 50 mL/min

Polishing time: 5 min

Working pressure: 4.9 kPa (50 g/cm²)

<Evaluation of polishing performance>

Polishing rate: calculated in terms of a decrease in weight of the aluminum disk after polishing

Surface roughness: measured using Talystep and Talydata 2000 (products of Rank Taylor Hobson Co.)

5 Depth of a scratch caused by polishing: obtained by analyzing the shape of the scratches using a probe-type surface analyzer (P-12, product of TENCOR Corporation) in a three dimensional mode.

10 Table 2 shows the evaluation results of the polishing performance. In Table 2, scratch score "A" denotes scratches caused by polishing having a depth of 2 nm or less, and scratch score "B" denotes scratches caused by polishing having a depth of 2 to 10 nm. No scratches having a depth of 10 nm or more were caused in
15 the Examples or in the Comparative Examples.

20 Surface cleanness (bright points): the number of bright points (bright points, abrasive deposits, abrasive dust, chemical agent deposits, etc.) on the side and backside of each disk were observed crosswise under a microscope in a dark vision field mode (product of Nikon, differential interference type, $\times 50$). Rating "A" was assigned when the total number of bright points is 20 or less, and "B" was assigned when the total number of bright points is 21 or more.

25 <Comparative Examples 1 to 3 and 5 to 6>

30 Water, inorganic acid 1, inorganic acid 2 and/or an organic phosphonic acid chelate compound, and an oxidizing agent were added to colloidal silica (Syton HT-50, product of Du Pont Kabushiki Kaisha) in the proportions shown in Table 2, to thereby prepare aqueous polishing compositions. Polishing was performed in the same manner as employed in the Examples. The results are shown in Table 3.

<Comparative Example 4>

35 Water, aluminum nitrate, and hydrogen peroxide were added to colloidal silica (Syton HT-50, product of Du Pont Kabushiki Kaisha) in the proportions shown in Table

2, to thereby prepare an aqueous polishing composition. Polishing was performed in the same manner as employed in the Examples. The results are shown in Table 3.

<Comparative Example 7>

5 White carbon (E-150J, product of Nippon Silica Industrial Co., Ltd.) was ground in a media agitating mill, and coarse particles were removed through classification, whereby silicon oxide particles having a mean particle size of 0.1 μm were produced.

10 Subsequently, water, inorganic acid 1, inorganic acid 2, and an oxidizing agent were added to the silicon oxide particles in the respective proportions shown in Table 2, to thereby prepare a variety of aqueous polishing compositions. Polishing was performed in the same manner

15 as employed in the Examples. The results are shown in Table 3.

<Comparative Example 8>

 Titanium oxide (Supertitania F-4, product of Showa Titanium Co., Ltd.) was ground by a media agitating mill,

20 and coarse particles were removed by classification, whereby titanium oxide particles having a mean particle size of 0.2 μm were produced. Subsequently, water and aluminum nitrate were added to the titanium oxide particles in the respective proportions shown in Table 2,

25 to thereby prepare an aqueous polishing composition. Polishing was performed in the same manner as employed in the Examples. The results are shown in Table 3.

<Comparative Examples 9 and 10>

 Each of alumina and zirconia was ground by a media

30 agitating mill, and coarse particles were removed by classification, whereby alumina particles and zirconia particles having a mean particle size of 0.2 μm were produced. Subsequently, water, inorganic acid 1, inorganic acid 2 or an organic phosphonic acid chelate

35 compound, and an oxidizing agent were added to the respective oxide particles in the respective proportions

shown in Table 2, to thereby prepare aqueous polishing compositions. Polishing was performed in the same manner as employed in the Examples. The results are shown in Table 3.

5

Table 1

Abrasive grains	Trade name	Mean particle size [μm]
Silicon oxide (silica)	HT-50	0.05
Silicon oxide (silica 2)	E-150J/AEROSIL 50	0.1/0.1
Titanium oxide (titania)	F-4	0.2
Aluminum oxide (alumina)	—	0.2
Zirconium oxide (zirconia)	—	0.2

Table 2 (1/2)

Ex.	Polishing agent Type	Polishing accelerator 1		Polishing accelerator 2		Oxidizing agent		Surface cleaning agent	
		Amount %	Type	Amount %	Type	Amount %	Type	Type	Amount %
1	Silica	10	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	EG	1.0
2	Silica	10	Phosphoric acid	1.5	Nitric acid / PBTC	0.4/1.0	H ₂ O ₂	EG	1.0
3	Silica	10	Phosphonic acid	3.0	HEDP	2.0	H ₂ O ₂	EG	1.0
4	Silica	10	Phosphonic acid	1.5	Nitric acid	0.4	H ₂ O ₂	EG	1.0
5	Silica	10	Phosphoric acid	1.5	NTMP	2.0	H ₂ O ₂	DEG	1.0
6	Silica 2	10	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	EG	1.0
7	Silica 2	10	Phosphonic acid	1.5	Nitric acid	0.4	H ₂ O ₂	EG	1.0
8	Titania	6	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	EG	1.0
9	Alumina	15	Phosphoric acid	1.5	HEDP	2.0	H ₂ O ₂	EG	1.0
10	Zirconia	15	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	EG	1.0

Table 2 (2/2)

Ex.	pH	Polishing rate [$\mu\text{m}/\text{min}$]	Surface roughness Ra[Å]	Scratches	Protrusions, Pits	Bright points
1	1.3	0.18	2	A	A	A
2	1.2	0.22	2	A	A	A
3	1.4	0.20	2	A	A	A
4	1.4	0.21	2	A	A	A
5	1.5	0.20	2	A	A	A
6	1.4	0.20	2	A	A	A
7	1.4	0.20	2	A	A	A
8	1.4	0.21	3	A	A	A
9	1.4	0.21	3	A	A	A
10	1.4	0.20	3	A	A	A

Table 3 (1/2)

Comp. Ex.	Polishing agent		Polishing accelerator 1		Polishing accelerator 2		Oxidizing agent		Surface cleaning agent	
	Type	Amount %	Type	Amount %	Type	Amount %	Type	Amount %	Type	Amount %
1	Silica	10	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	0.5	-	-
2	Silica	10	Phosphoric acid	1.5	Nitric acid / PBTC	0.4/1.0	H ₂ O ₂	0.5	-	-
3	Silica	10	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	0.5	-	-
4	Silica	10	Aluminum nitrate	5.0	-	-	H ₂ O ₂	1.0	-	-
5	Silica	10	Phosphonic acid	1.5	Nitric acid	0.4	H ₂ O ₂	0.5	-	-
6	Silica	10	Phosphoric acid	1.5	NTMP	2.0	H ₂ O ₂	0.5	-	-
7	Silica 2	10	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	0.5	-	-
8	Titania	6	Aluminum nitrate	5.0	-	-	-	-	-	-
9	Alumina	15	Phosphoric acid	1.5	HEDP	2.0	H ₂ O ₂	0.5	-	-
10	Zirconia	15	Phosphoric acid	1.5	Nitric acid	0.4	H ₂ O ₂	0.5	-	-

Table 3 (2/2)

Comp. Ex.	pH	Polishing rate [$\mu\text{m}/\text{min}$]	Surface roughness Ra[Å]	Scratches	Protrusions, Pits	Bright points
1	1.3	0.18	2	A	A	B
2	1.2	0.22	2	A	A	B
3	1.4	0.19	2	A	A	B
4	2.0	0.08	4	B	B	B
5	1.4	0.21	2	A	A	B
6	1.5	0.20	2	A	A	B
7	1.4	0.20	2	A	A	B
8	3.0	0.21	3	B	B	B
9	1.4	0.21	3	A	A	B
10	1.4	0.20	3	A	A	B

INDUSTRIAL APPLICABILITY

By polishing a disk through use of the polishing composition of the present invention, a high-quality polished surface having a remarkably low surface roughness and no protrusions or deposits can be obtained at high polishing rate. In addition, a magnetic disk made of the thus-polished disk is useful as a hard disk of low-flying-height-type capable of high-density recording. In particular, the magnetic disk made of the polished disk is advantageously utilized as a high recording density medium (having a recording density of 3 Gbits/inch² or more), as represented by a medium for MR head employing a magnetoresistance effect. Furthermore, a medium having less recording density which has been produced by use of the composition of the invention is also useful because of high reliability.

CLAIMS

1. A polishing composition characterized by comprising water, abrasive grains, a polishing accelerator, and a surface cleaning agent which is a
5 compound comprising a main chain having 2 to 6 carbon atoms and optionally an oxygen atom, said compound having a plurality of hydroxyl groups.

2. The polishing composition according to claim 1, wherein the surface cleaning agent is at least one
10 species selected from the group consisting of glycerin, ethylene glycol, diethylene glycol, triethylene glycol, and propylene glycol.

3. The polishing composition according to claim 1 or 2, wherein the composition contains the surface
15 cleaning agent in an amount of 0.1 to 15 mass% based on the entirety of the polishing composition.

4. The polishing composition according to any one of claims 1 to 3, wherein the abrasive grains are at least one species selected from the group consisting of
20 alumina, titania, silica, and zirconia.

5. The polishing composition according to any one of claims 1 to 4, wherein the abrasive grains have a mean particle size falling within a range of 0.001 to 0.5 μm .

6. The polishing composition according to any one
25 of claims 1 to 5, wherein the abrasive grains are colloid particles.

7. The polishing composition according to any one of claims 1 to 6, wherein the polishing accelerator is at least one species selected from the group consisting of a
30 phosphorus-containing inorganic acid or a salt thereof; a not-phosphorus-containing inorganic acid, or a salt of the second inorganic acid; an organic phosphonic acid chelate compound; and an oxidizing agent.

8. The polishing composition according to any one
35 of claims 1 to 7, wherein the polishing composition contains, as a polishing accelerator, (i) a phosphorus-containing inorganic acid or a salt thereof; (ii) a not-

phosphorus-containing inorganic acid, or a salt of the not-phosphorus-containing inorganic acid and/or an organic phosphonic acid chelate compound; and (iii) an oxidizing agent.

5 9. The polishing composition according to claim 7 or 8, wherein the phosphorus-containing inorganic acid is phosphoric acid or phosphonic acid.

10 10. The polishing composition according to any one of claims 7 to 9, wherein the not-phosphorus-containing inorganic acid is at least one species selected from the group consisting of nitric acid, sulfuric acid, amidosulfuric acid, and boric acid.

15 11. The polishing composition according to any one of claims 7 to 10, wherein the oxidizing agent is a peroxide or a nitrate salt.

12. The polishing composition according to claim 11, wherein the peroxide is at least one species selected from the group consisting of hydrogen peroxide, a perborate salt, and a persulfate salt.

20 13. The polishing composition according to any one of claims 1 to 12, which has a pH falling within a range of 1 to 5.

25 14. The polishing composition according to any one of claims 7 to 13, wherein the amount of abrasive grains falls within a range of 3 to 30 mass%.

15. The polishing composition according to any one of claims 7 to 14, wherein the amount of inorganic acid or a salt thereof falls within a range of 0.1 to 8 mass%.

30 16. The polishing composition according to any one of claims 7 to 15, wherein the amount of organic phosphonic acid chelate compound falls within a range of 0.01 to 10 mass%.

35 17. The polishing composition according to any one of claims 7 to 16, wherein the amount of oxidizing agent falls within a range of 0.2 to 5 mass%.

18. The polishing composition according to any one of claims 7 to 13, wherein the amount of abrasive grains

falls within a range of 3 to 30 mass%, the amount of inorganic acid or a salt thereof falls within a range of 0.1 to 8 mass%, the amount of organic phosphonic acid chelate compound falls within a range of 0.01 to 10 mass%, and the amount of oxidizing agent falls within a range of 0.2 to 5 mass%.

19. The polishing composition according to any one of claims 1 to 18, further containing a surfactant.

20. The polishing composition according to any one of claims 1 to 19, further containing an anticorrosive.

21. The polishing composition according to any one of claims 1 to 20, further containing a preservative.

22. The polishing composition according to any one of claims 1 to 21, further containing an anti-gelling agent.

23. A kit of two compositions for forming the polishing composition as set forth in any one of claims 1 to 22 by mixing or by mixing and diluting the two compositions.

24. The kit according to claim 23, in which one of said two compositions comprises abrasive grains and water, and the other one comprises an inorganic acid, an oxidizing agent and a surface cleaning agent.

25. The kit according to claim 23 or 24, for transportation or storage.

26. A composition which forms the polishing composition according to any one of claims 1 to 22 by dilution.

27. A method of employing the composition as set forth in any one of claims 23 to 26 as a composition for transportation and storage.

28. A method of polishing a magnetic disk substrate, the method employing a polishing composition as recited in any one of claims 1 to 26.

29. A method for preparing a polishing composition by mixing the two compositions of the kit as set forth in any one of claims 23 to 25.

30. A method for polishing characterizing by mixing the two compositions of the kit as set forth in any one of claims 23 to 25 and employing the obtained composition for polishing.

5 31. A method for polishing characterizing by preparing a composition having a higher concentration than that for polishing, diluting said prepared composition to obtain the polishing composition as set forth in any one of claims 1 to 22, and employing the
10 obtained composition for polishing.

32. A method of producing a magnetic disk substrate, the method employing the method as recited in any one of claims 28, 30 and 31.

33. The method according to claim 32, in which the
15 magnetic disc substrate has a recording density of 3 Gbit/inch² or more.

34. The method according to claim 32 or 33, in which the magnetic disc substrate is an aluminum substrate.

20 35. The method according to claim 32 or 33, in which the magnetic disc substrate is a NiP-plated aluminum substrate.