

METHOD OF PRETREATING A NONMAGNETIC SUBSTRATE AND A MAGNETIC RECORDING MEDIUM

CROSS-REFERENCE TO RELATED APPLICATION

- 5 This application is based on, and claims priority to, Japanese Application No. 2003-421604, filed on December 18, 2003.

BACKGROUND OF THE INVENTION

10 Technical Field of the Invention

 The present invention relates to a method of pretreating for a nonmagnetic substrate, for example, to a method to form a Ni-P layer by electroless plating on a glass substrate that is a nonmagnetic substrate used in manufacturing a magnetic disk, in particular, to a method of pretreatment for the plating.

- 15 The invention also relates to a magnetic recording medium manufactured by employing such a method and mounted on magnetic recording apparatuses including external storage devices for a computer, in particular a hard disk device.

20 Description of the Related Art

 Aluminum alloys have been used for a material of conventional nonmagnetic substrate. Recently, hard disk devices are proceeding directing to large capacity, small size, and light weight. Accordingly, a magnetic disk is required flatter, having a smaller diameter, and thinner than conventional ones.

- 25 Since a conventional substrate of an aluminum alloy is difficult to cope with these demands of the market, glass is increasingly used for a substrate material.

It is technologically difficult to form a Ni-P layer on a glass substrate by an electroless plating method enough adhesively, homogeneously, and smoothly. To form a Ni-P layer having good adhesivity, sufficient homogeneity, and smoothness to obtain a well performed magnetic disk, various methods of pre- and post- treatments have been proposed for the electroless plating process.

The treatment methods include a method (disclosed in Japanese Unexamined Patent Application Publication No. H1-176079, for example) in which electroless plating is conducted after a treatment in an aqueous solution containing palladium chloride and stannous chloride, followed by a treatment in a solution of alkali carbonate, a solution of alkali hydrogencarbonate, or a solution of a mixture of the two compounds. In another method (disclosed in Japanese Unexamined Patent Application Publication No. S53-19932, for example), electroless plating is conducted after a series of pretreatments including two-stage etching with a chromic acid - sulfuric acid mixed solution and with a nitric acid solution, etching with a strong alkaline solution, sensitizing treatment with dilute stannous chloride, and activation treatment with silver salt solution and palladium salt solution. Still another method is proposed (in Japanese Unexamined Patent Application Publication No. S48-85614, for example) in which electroless plating is conducted after pretreatments including cleaning with warm liquid of sulfuric acid and potassium bichromate, sensitization with stannous chloride made acidic by hydrochloric acid, and activation with palladium chloride solution. Also proposed is a method in which electroless plating is conducted after pretreatments of alkaline degreasing, etching with hydrofluoric acid, sensitizing with stannous chloride solution, and activation with palladium chloride solution.

Further, a method is disclosed (in Japanese Unexamined Patent Application

Publication No. H7-334841, for example) in which electroless plating is conducted after processes of: adequate degreasing of the glass substrate, etching to enhance anchoring effect, removing contaminants adhered on the substrate surface during the etching process, chemically homogenizing the substrate surface by surface modulation process, sensitizing treatment, and activation treatment. Preferable materials disclosed in the reference are an aqueous solution containing hydrofluoric acid and potassium hydrofluoride for etching, hydrochloric acid for removing surface contaminants, and an aqueous solution containing sodium methoxide for surface modulation.

10 A method (disclosed in Japanese Unexamined Patent Application Publication No. 2000-163743, for example) conducts pretreatments on the surface of a glass substrate including sequentially degreasing, etching, warm pure water treatment, silane coupling agent treatment, activation treatment, and acceleration treatment, and then carry out electroless plating, and a heating treatment follows. Preferable materials disclosed in the reference are amino silane coupling agent for the silane coupling agent, aqueous solution of palladium chloride for activator, and aqueous solution of sodium hypophosphite for accelerator.

20 A Ni-P layer formed on a glass substrate according to any one of the above-described known methods, however, have not been possible to achieve satisfactory thickness (of one to several microns) and adhesiveness, homogeneity, and smoothness at that thickness, which are required to obtain a magnetic disk having a well-performed magnetic recording layer.

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SUMMARY OF THE INVENTION

Therefore, an object of the present invention is to provide a method of pretreatment for a nonmagnetic substrate allowing forming a layer that includes
5 a Ni-P plating and exhibits a plating layer thickness, adhesiveness, homogeneity, and smoothness so that a well-performed magnetic recording layer is provided on a nonmagnetic substrate of glass. Another object of the invention is to provide a magnetic recording medium produced employing such a method.

The present invention provides a method of pretreatment prior to forming a
10 magnetic recording layer on a nonmagnetic substrate. The method comprises steps of cleaning a surface of the substrate, forming Ni layer on the surface of the nonmagnetic substrate after cleaning, forming a palladium layer that is a catalyst layer on the Ni layer, and forming a Ni-P plating layer on the palladium layer.

15 Advantageously, the substrate is a glass substrate and the step of cleaning the substrate surface includes a step of degreasing the substrate surface and a step of activating the substrate surface; the step of forming the Ni layer includes a step of applying a nickel chelate agent or a nickel soap agent, a step of metallizing baking the nickel chelate agent or the nickel soap agent, and a step
20 of activating the metallized Ni layer; the step of forming the palladium layer includes a step of converting the palladium layer to a catalyst layer; and the step of plating the Ni-P plating layer includes a step of electroless plating the Ni-P plating layer and a following step of heating treatment.

Advantageously, the step of forming the Ni layer includes a step of baking
25 a nickel chelate agent or a nickel soap agent to form a nickel metallic film; the step of forming the palladium layer includes a step of bonding palladium with a

surface of the nickel metallic film; and the step of forming the Ni-P plating layer is carried out by electroless plating after the step of bonding, and includes a subsequent step of heating treatment.

Preferably, surface roughness of the substrate surface after the pretreatment is at most 0.5 nm and micro surface waviness is at most 0.5 nm.

Preferably, the nickel chelate agent that is a material for the Ni layer has a structure represented by formula (1):



wherein i, j, and k are positive integers.

Preferably, the nickel soap agent that is another material for the Ni layer has a structure represented by one of $\text{Ni}(-\text{OOCCH}(\text{C}_n\text{H}_{2n+1})\text{C}_p\text{H}_{2p+1})_2$ and $\text{Ni}(-\text{OOC}\text{C}_m\text{H}_{2m+1})_2$ wherein m, n, and p each are a positive integer of one or greater.

Preferably, the step of metallizing baking is carried out in an inert gas atmosphere and at a temperature in the range of 250°C to 400°C.

Advantageously, the step of converting the palladium layer to a catalyst layer is a treatment of palladium chloride.

Preferably, the Ni-P plating layer contains phosphorus in a range of 1.0 wt% to 13.0 wt% and has a thickness at least 1.0 μm.

Preferably, the heating treatment after the electroless Ni-P plating is carried out at a temperature in a range of 250°C to 300°C for at least 1 hr.

The present invention also provides a magnetic recording medium comprising a nonmagnetic substrate, a magnetic recording layer formed above the substrate, and a pretreatment layer formed between the

substrate and the magnetic recording layer by the method of pretreating the substrate described above. The pretreatment layer comprises a nickel adhesion layer formed on the

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substrate, a palladium catalyst layer formed on the adhesion layer, and a Ni-P plating layer formed on the catalyst layer.

BRIEF DESCRIPTION OF DRAWINGS

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Fig. 1 is a flow chart illustrating a first aspect of the invention that is an outline of a method of pretreating a glass substrate as a nonmagnetic substrate,

Fig. 2 is a schematic sectional view of a magnetic recording medium manufactured employing the pretreatment method of Fig. 1, and

10 Fig. 3 is a flow chart illustrating the pretreatment method of Fig. 1 in detail.

DETAILED DESCRIPTION OF THE INVENTION

Some preferred embodiments of the invention will be described in detail in
15 the following with reference to accompanying drawings.

[First aspect of embodiment]

A first aspect of embodiment of the invention is described referring to Figs. 1 through 3.

(A method of pretreatment)

20 Steps S1 through S4 in Fig. 1 illustrate a method of pretreatment of a nonmagnetic substrate of glass. Steps S11 through S23 in Fig. 3 illustrate the method of pretreatment of Fig. 1 more in detail.

Step S1 is a step of cleaning a surface of the glass substrate 1.

In the step S1, the surface of the glass substrate 1 is degreased (steps S11
25 and S12) and activated (step S13).

Step S2 is a step of forming a nickel adhesion layer 2 on the surface of the

glass substrate 1 after cleaning.

In the step S2, a nickel chelate agent or a nickel soap agent is applied on the glass substrate 1 (step S14 or step S15); metallization baking is carried out to the applied nickel-containing agent to obtain a metallic nickel film (step S16 or
5 step S17); and the metallic nickel film is activated (step S18) obtaining a nickel adhesion layer 2.

Step S3 is a step of forming a palladium catalyst layer 3 on the nickel adhesion layer 2.

In the step S3, a palladium catalyst layer 3 is formed by converting
10 palladium to a catalyst (step S19) on the nickel adhesion layer 2.

Step S4 is a step of forming a Ni-P plating layer 4 on the palladium catalyst layer 3.

In the step S4, a Ni-P plating layer 4 is formed by conducting electroless Ni-P plating (steps S20 through S22) and followed by heating treatment (step
15 S23).

(Magnetic recording medium)

Fig. 2 illustrates a sectional structure of a magnetic recording medium 100 manufactured by employing the pretreatment method shown in Figs. 1 and 3.

The magnetic recording medium 100 comprises a glass substrate 1 that is a
20 nonmagnetic substrate with a disk shape, and layers sequentially laminated on the substrate including a nickel adhesion layer 2 having a thickness of 10 nm to 50 nm, a palladium catalyst layer 3 having a thickness of 1 nm to 10 nm, and a Ni-P plating layer 4 having a thickness of at least 1 μm . On the pretreatment layer including the layers 2 through 4, a desired magnetic recording layer 5 is
25 formed.

The magnetic recording layer 5 is formed through the following procedure.

After a Ni-P plating layer is formed on the surface of the glass substrate by an electroless plating method, the surface of the Ni-P plating 4 is subjected to a necessary smoothing treatment and a texturing treatment. A magnetic recording layer 5 is produced by sequentially forming a nonmagnetic metallic underlayer, a magnetic layer of a thin film of a ferromagnetic alloy, a protective layer, and a liquid lubricant layer on the pretreated surface.

A magnetic recording medium 100 manufactured by such a method is commonly used as a magnetic disk mounted on a hard disk drive, which is an external storage device of an information processing apparatus, e.g., a computer.

10 According to the invention, a well-performing magnetic disk can be manufactured comprising a Ni-P plating layer exhibiting enough thickness of one to several microns, and satisfactory adhesiveness, homogeneity, and smoothness at that thickness.

The following describes in detail each step in the pretreatment method shown in Fig. 3.

(Steps S11 through S13)

The surface of the glass substrate 1 is cleaned (steps S11 and S12) by degreasing the surface and removing organic coating layers and particles.

After cleaning, an activation treatment (step S13) is conducted on the surface of the glass substrate 1 by dipping in dilute acid solution to peel off inactive oxide films existing on the surface of the glass substrate 1 and to modify the functional groups on the surface of the glass substrate 1 into reactive silanol groups (Si-OH).

(Steps S14 through S18)

25 On the surface of the glass substrate 1 covered with reactive silanol groups (Si-OH), an appropriate amount of a nicked chelate agent (represented by

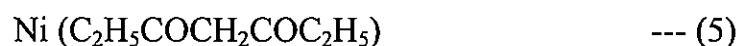
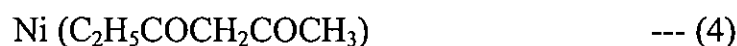
formula 1) or a nickel soap agent (represented by formula 2) is applied (steps S14 or S15). The agent becomes an adhesion layer material between the glass substrate 1 and the Ni-P plating layer 4.

The nickel chelate agent for the adhesion layer material is represented by
5 general formula (1),

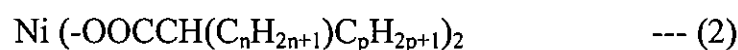


wherein i, j, and k are positive integers.

The nickel chelate agent is preferably, for example, nickel acetylacetonate represented by formula (3), nickel propioneacetonate represented by formula (4),
10 nickel propioneethylate represented by formula (5), or a mixture of the substances of formulas (3), (4), and (5).



15 The nickel soap agent, which is another material for the adhesion layer, is represented by a general formula (2) and (2'),

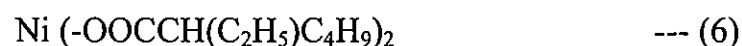


wherein n and p are a positive integer of one or larger.



20 wherein m is a positive integer of one or larger.

The nickel soap agent is preferably nickel 2-ethylhexanoate represented by formula (6) or nickel stearate represented by formula (7), for example.



25 In the subsequent process of baking at an appropriate temperature under an inert gas atmosphere, the nickel chelate agent or the nickel soap agent is

thermally modified. Organic compounds are decomposed and volatilized; and a metallic component nickel makes substitution reaction with the silanol group previously modulated and generated on the surface of the glass substrate 1, obtaining a nickel metallic film composed of dehydrogenated nickel silanoxide (Si-ONi) (steps S16 or S17).

The nickel metallization baking treatment is preferably conducted at a temperature in the range of 250°C to 350°C for the nickel chelate agent, while at a temperature in the range of 300°C to 400°C for the nickel soap agent, for at least 30 min.

Subsequent activation treatment for the nickel metallic film peels off a nickel oxide film that forms outermost several nanometer layer of the nickel metallic film by means of an acid treatment (step S18).

(Step S19)

A palladium catalyst generating treatment is appropriately conducted to provide a catalyst layer for precipitation of Ni-P plating.

(Steps S20 through S23)

Electroless Ni-P plating is conducted under predetermined conditions.

A plating solution to form the Ni-P plating layer 4 can be selected from every Ni-P plating solution exhibiting nonmagnetic or soft magnetic property with a phosphorus concentration in the range of 1 to 13 wt%. Specific examples of the plating solutions include: so-called nonmagnetic high phosphorus concentration type Ni-P plating solution (phosphorus concentration from 11 to 13 wt%, for example NIMUDEN HDX, a product of C.Uyemura & Co., Ltd.), nonmagnetic or soft magnetic, medium phosphorus concentration type Ni-P plating solution (phosphorus concentration from 6 to 8 wt%, for example MELPLATE NI-867, a product of Meltex Inc., and phosphorus concentration

from 3 to 6 wt%, for example MELPLATE NI-802, a product of Meltex Inc.), and soft magnetic low phosphorus concentration type Ni-P plating solution (phosphorus concentration from 1 to 2 wt%, for example NIMUDEN LPY, a product of C.Uyemura & Co., Ltd., and TOP NICORON LPH, a product of
5 Okuno Chemical Industries Co., Ltd.).

The Ni-P plating layer can also be formed using a higher phosphorus concentration type Ni-P plating solution with phosphorus concentration higher than 14 wt% that is commercially available and facilitates precipitation of the plating.

10 Various types of Ni-P plating is employed, depending on each purpose, including the nonmagnetic high phosphorus concentration Ni-P plating, the nonmagnetic or soft magnetic medium phosphorus concentration Ni-P plating, and the soft magnetic low phosphorus concentration Ni-P plating, as described below.

15 The nonmagnetic high phosphorus concentration Ni-P plating layer formed on a nonmagnetic glass substrate allows:

(1) to obtain exact smoothness for attaining high density recording that is difficult with high rigidity glass substrate;

(2) to manufacture an anisotropically aligned medium employing LZT (laser
20 zone texturing) or tape texturing; and

(3) to enhance coercivity of a magnetic film by applying a bias voltage on the substrate in the sputtering process.

The soft magnetic low phosphorus concentration Ni-P plating film formed on a nonmagnetic glass substrate can be used for a soft magnetic backing layer
25 of a perpendicular magnetic recording medium. A perpendicular magnetic recording medium exhibiting high recording density can be obtained by a double

layer perpendicular magnetic recording medium in which soft magnetic film called a soft magnetic backing layer is provided under a magnetic recording layer that carries information recording function. The backing layer has high saturation magnetic flux density and facilitates flow of magnetic flux generated
5 by the magnetic head.

The nonmagnetic or soft magnetic, medium phosphorus concentration Ni-P plating layer formed on a nonmagnetic glass substrate can be used for an underlayer plating film (striking plating) that is an adhesion layer between a low phosphorus concentration Ni-P plating film and the nonmagnetic substrate, as
10 well as applications that use the high phosphorus concentration Ni-P plating film and the low phosphorus concentration Ni-P plating film.

Subsequent heating treatment (at 250°C for 4 hr) completes forming an electroless Ni-P plating layer 4 having a thickness at least 1.0 μm that exhibits excellent adhesiveness, homogeneity, and smoothness.

15 This heating treatment after the electroless Ni-P plating is preferably conducted at a temperature in the range of 250°C to 300°C for at least 1 hr in an inert gas atmosphere.

Degree of surface roughness R_a of the glass substrate 1 affects physical anchoring effect on adhesiveness of the Ni-P plating layer 4. For surface
20 roughness R_a larger than 0.5 nm, there is certain expectation of enhancement of adhesiveness of the Ni-P plating layer 4. For surface roughness R_a smaller than 0.5 nm, physical anchoring effect is hardly expected on an ultra smooth glass substrate 1. Nevertheless, the method of the invention allows such an ultra smooth glass substrate 1 with the surface roughness smaller than 0.5 nm to
25 exhibit satisfactory adhesiveness of the Ni-P plating layer 4.

As described previously, a strong chemical bond is developed between the

glass substrate 1 and the nickel adhesion layer 2 at the interface on the glass substrate 1 by generation of the dehydrogenized nickel silanoxide (Si-ONi) from the silanol group (Si-OH) on the glass substrate 1 and the nickel chelate agent or the nickel soap agent that is a material of the nickel adhesion layer 2. The
5 chemical bond assures sufficient adhesiveness of the Ni-P plating layer 4 even on an ultra smooth glass substrate 1.

A Ni-P plating layer 4 formed on a glass substrate 1 obtained according to the method of the invention has an enough thickness (in the range of 1 μm to 5 μm) and exhibits satisfactory adhesiveness, homogeneity, and smoothness at
10 that thickness range, which are necessary to obtain a magnetic disk having an excellent magnetic recording layer 5, using every Ni-P plating solution exhibiting nonmagnetic or soft magnetic property.

[Second aspect of embodiment]

The second aspect of embodiment of the invention is described referring to
15 Table 1 and Table 2. The same parts as in the first aspect of embodiment are given the same symbol, and description is omitted.

This second embodiment describes Examples and Comparative Examples that are specific examples of pretreatment methods for the glass substrate 1.

Table 1 shows treatment conditions for each treatment step. Treatment
20 items in Table 1 are treatment steps, treatment liquid, concentration, temperature, treatment time, and the other. The treatment steps (1) through (9) correspond to the steps S11 through S23 in Fig. 3.

Table 1

treatment step	treatment liquid	concentration (wt%)	temperature (°C)	time	other
(1) detergent degreasing	alkali detergent	1.5	50	3 min	
(2) alkali degreasing	KOH	7.5	50	3 min	
(3) glass activation	HF + NH ₃ F	1.0	20	3 min	
(4) application of Ni chelate agent or Ni soap agent	Ni acetylacetonate /toluene	0.3	20	-	
	Ni 2-ethyl hexanate /cyclohexane	0.3	20	-	
(5) Ni metallizing baking	Ni acetylacetonate	-	300	1 hr	in N ₂
	Ni 2-ethyl hexanate	-	380	1 hr	in N ₂
(6) activation of Ni metallic film	HNO ₃ solution	30	20	2 min	
(7) formation of Pd catalyst layer	PdCl ₂ + NaOH	3.0	20	3min	
(8) Ni-P plating	NIMUDENN HDX	-	80	20 min	
	MELPLATE NI-867	-	70	35 min	
	NIMUDEN LPY	-	80	25 min	
(9) heating		-	250	4 hr	in N ₂

The following describes specific examples conducted under various treatment conditions.

5 (Example 1)

Description is made on Example 1.

A chemically reinforced glass substrate was used for a glass substrate 1. A Ni-P plating layer 4 indicated in Fig. 2 was formed by means of an electroless plating method through the treatment steps (1) through (9) on the substrate surface under the conditions given below.

(1) Detergent degreasing: detergent concentration 1.5 wt%, a treatment at 50°C for 3 min

(2) Alkali degreasing: KOH concentration 7.5 wt%, a treatment at 50°C for 3 min

- (3) Glass surface activation: HF + NH₃F concentration 1.0 wt%, a treatment at 20°C for 3 min
- (4) Application of nickel chelate agent, a material for nickel adhesion layer: application by dipping in nickel acetylacetonate / toluene solution with nickel acetylacetonate concentration 0.3 wt%
- (5) Baking for nickel metallized film: a baking treatment in nitrogen gas atmosphere at 300°C for 1 hr
- (6) Activation of nickel metallized film: HNO₃ concentration 30 wt%, a treatment at 20°C for 2 min
- 10 (7) Formation of palladium catalyst layer: concentration of PdCl₂ + NaOH 3.0 wt%, a treatment at 20°C for 3 min
- (8) Formation of Ni-P plating layer: plating solution NIMUDEN HDX (phosphorus concentration 15 - 20 wt% produced by C.Uyemura & Co., Ltd., a treatment at 80°C for 20 min (Ni-P plating layer thickness 3.0 μm)
- 15 (9) Heating of Ni-P plating layer: a heating treatment in nitrogen gas atmosphere at 250°C for 4 hr
- (Example 2)

Description is made on Example 2.

- A Ni-P plating layer 4 was formed by means of an electroless plating method through the same treatment steps and under the same conditions as in
- 20 Example 1 except the following step and condition.

- (8) Formation of Ni-P plating layer 4: plating solution MELPLATE NI-867 (phosphorus concentration 6 - 8 wt% produced by Meltex Inc., a treatment at 70°C for 35 min (Ni-P plating layer thickness 3.0 μm)
- 25 (Example 3)

Description is made on Example 3.

A Ni-P plating layer 4 was formed by means of an electroless plating method through the same treatment steps and under the same conditions as in Example 1 except the following step and condition.

- (8) Formation of Ni-P plating layer: plating solution NIMUDEN LPY
5 (phosphorus concentration 1 - 2 wt% produced by C.Uyemura & Co., Ltd.), a treatment at 80°C for 25 min (Ni-P plating layer thickness 3.0 μm).

(Example 4)

Description is made on Example 4.

- A Ni-P plating layer 4 was formed by means of an electroless plating
10 method through the same treatment steps and under the same conditions as in Example 1 except the following steps and conditions.

- (4) Application of nickel soap agent, a material for nickel adhesion layer: application by dipping in nickel 2-ethyl hexanate / cyclohexane solution with nickel 2-ethyl hexanate concentration 0.3 wt%,
15 (5) Baking for nickel metallized film: a baking treatment in nitrogen gas atmosphere at 380 °C for 1 hr,
(8) Formation of Ni-P plating layer: plating solution MINUDENN HDX (phosphorus concentration 11 - 13 wt%, produced by C.Uyemura & Co., Ltd.), a treatment at 80 °C for 20 min (Ni-P plating layer thickness 3.0 μm).

20 (Example 5)

Description is made on Example 5.

- A Ni-P plating layer 4 was formed by means of an electroless plating method through the same treatment steps and under the same conditions as in Example 1 except the following steps and conditions.
25 (4) Application of nickel soap agent, a material for nickel adhesion layer: application by dipping in nickel 2-ethyl hexanate / cyclohexane solution with

nickel 2-ethyl hexanate concentration 0.3 wt%,

(5) Baking for nickel metallized film: baking treatment in nitrogen gas atmosphere at 380 °C for 1 hr,

(8) Formation of Ni-P plating layer: plating solution MELPLATE NI-867

5 (phosphorus concentration 6 - 8 wt% produced by Meltex Inc.), a treatment at 70°C for 35 min (Ni-P plating layer thickness 3.0 μm)

(Example 6)

Description is made on Example 6.

A Ni-P plating layer 4 was formed by means of an electroless plating
10 method through the same treatment steps and under the same conditions as in Example 1 except the following steps and conditions.

(4) Application of nickel soap agent, a material for nickel adhesion layer: application by dipping in nickel 2-ethyl hexanate / cyclohexane solution with nickel 2-ethyl hexanate concentration 0.3 wt%,

15 (5) Baking for nickel metallized film: baking treatment in nitrogen gas atmosphere at 380 °C for 1 hr,

(8) Formation of Ni-P plating layer: plating solution NIMUDEN LPY (phosphorus concentration 1 - 2 wt% produced by C.Uyemura & Co., Ltd.), a treatment at 80°C for 25 min (Ni-P plating layer thickness 3.0 μm).

20 Comparative examples to Examples 1 through 6 shown above are described below.

(Comparative Example 1)

Description is made on Comparative Example 1.

A chemically reinforced glass substrate was used for a glass substrate 1.

25 A Ni-P plating layer was formed by means of an electroless plating method through the treatment steps (1) through (7) on the substrate surface under the

conditions shown below.

- (1) Detergent degreasing: detergent concentration 1.5 wt%, a treatment at 50°C for 3 min,
- (2) Alkali degreasing: KOH concentration 7.5 wt%, a treatment at 50°C for 3 min,
- (3) Roughening of glass surface: chromic acid plus sulfuric acid concentrations 40 wt% plus 40 wt%, a treatment at 60°C for 10 min,
- (4) Catalyst treatment for catalyst provision: a treatment at 20°C for 30 min in an aqueous solution of a mixture of 0.3 g/L of PdCl_2 plus 15 g/L of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ plus 200 mL/L of 36 % HCl,
- (5) Acceleration treatment for catalyst provision: a treatment at 50°C for 5 min in an aqueous solution of 100 g/L of H_2SO_4 ,
- (6) Formation of Ni-P plating layer 4: plating solution NIMUDEN HDX (phosphorus concentration 15 - 20 wt%, produced by C.Uyemura & Co., Ltd.), a treatment at 80°C for 3.0 min (Ni-P plating layer thickness 0.4 μm ; film deposition thicker than 0.4 μm was impossible due to peeling occurred during the plating process.),
- (7) Heating of Ni-P plating layer: a treatment in nitrogen atmosphere at 250°C for 4hr.

20 (Comparative Example 2)

Description is made on Comparative Example 2.

A Ni-P plating layer 4 was formed by means of an electroless plating method through the same treatment steps and under the same conditions as in Comparative Example 1 except the following step and condition.

- 25 (6) Formation of Ni-P plating layer: plating solution MELPLATE NI-867 (phosphorus concentration 6 - 8 wt%, produced by Meltex Inc.), a treatment at

80°C for 9.0 min (Ni-P plating layer thickness 0.7 μm; film deposition thicker than 0.7 μm was impossible due to peeling occurred during the plating process.).
(Comparative Example 3)

Description is made on Comparative Example 3.

5 A Ni-P plating layer was formed by means of an electroless plating method through the same treatment steps and under the same conditions as in Comparative Example 1 except the following step and condition.

(6) Formation of Ni-P plating layer: plating solution NIMUDEN LPY (phosphorus concentration 1 - 2 wt%, produced by C.Uyemura & Co., Ltd.), a
10 treatment at 80°C for 3.0 min (Ni-P plating layer thickness 0.5 μm; film deposition thicker than 0.5 μm was impossible due to peeling occurred during the plating process.).

Table 2 shows the evaluation results on Examples 1 through 6 and Comparative Examples 1 through 3. Evaluation items in Table 2 are adhesion
15 layer, plating layer, plating film thickness, adhesive force, and surface roughness (Ra). The marks ○ and X indicate sufficient adhesive force and insufficient adhesive force, respectively.

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Table 2

	adhesion layer	plating layer	plating layer thickness (μm)	adhesive force	surface roughness (nm)
Ex 1	Ni chelate	NIMUDEN HDX	3.0	○	0.32
Ex 2	Ni chelate	MELPLATE NI-867	3.0	○	0.33
Ex 3	Ni chelate	NIMUDEN LPY	3.0	○	0.30
Ex 4	Ni soap	NIMUDEN HDX	3.0	○	0.35
Ex 5	Ni soap	MELPLATE NI-867	3.0	○	0.36
Ex 6	Ni soap	NIMUDEN LPY	3.0	○	0.34
CEx 1	Sn / Pd	NIMUDEN HDX	0.4	X	0.56
CEx 2	Sn / Pd	MELPLATE NI-867	0.7	X	0.57
CEx 3	Sn / Pd	NIMUDEN LPY	0.5	X	0.61

Actual evaluation of adhesiveness of the Ni-P plating layer 4 was carried out according to the “Cross-cut Peeling Test” (Japanese Industrial Standards (JIS) K 5400 6.15) on the glass substrates 1 on which Ni-P plating layer is plated by an electroless plating method.

Mean surface roughness Ra was measured using an atomic force microscope (AFM) on the glass substrate 1 before and after plating. The mean surface roughness Ra before plating was 0.25 nm.

As shown in Table 2, the thickness of the Ni-P plating layer 4 formed on the glass substrate 1 by electroless plating attained 3.0 μm on the layers that were fabricated in Examples 1 through 6 by the treatment steps and conditions according to the invention shown in Table 1. Table 2 also confirms that the Ni-P plating layers 4 in the Examples exhibit sufficient adhesivity in the range from low phosphorus concentration to high phosphorus concentration.

Surface roughness Ra was smaller than or equal to 0.5 nm and micro

surface waviness W_a was smaller than or equal to 0.5 nm after the plating. It has been confirmed that the increase of surface roughness is insignificant and the surface roughness is sufficiently held within the range required by magnetic disks.

5 In contrast, the thickness of the Ni-P plating layer 4 formed on the glass substrate 1 by electroless plating fell in the range of 0.4 to 0.7 μm on the layers that were fabricated in Comparative Examples 1 through 3 according to the known technology. It has been shown that a thick film not smaller than 1.0 μm is impossible and adhesiveness to a glass substrate 1 is significantly degraded.

10 As described so far, the method of the invention for forming a Ni-P plating layer comprises steps of degreasing and glass activation treatments on a glass substrate surface, application treatment of a nickel chelate agent or a nickel soap agent on the glass substrate surface, metallizing baking treatment of the nickel-containing agent, activation treatment of the nickel metallized film to form a
15 nickel adhesion layer 2, treatment for forming a palladium catalyst layer 3 on the nickel adhesion layer 2, electroless Ni-P plating in the phosphorus concentration range of 1 to 13 wt% on the palladium catalyst layer 3 to form a Ni-P plating layer 4, and a heating treatment. Owing to these featured treatment processes according to the invention, the Ni-P plating layer formed by electroless plating
20 has a thickness at least 1.0 μm and exhibits sufficient adhesivity, homogeneity, and smoothness. Therefore, a magnetic disk can be manufactured that is provided with a well-performed magnetic recording layer on a glass substrate.

 A magnetic recording medium according to the invention is manufactured by the method comprising the above-described pretreatment processes on the
25 nonmagnetic substrate and comprises a pretreatment layer including a nickel adhesion layer, a palladium catalyst layer, and a Ni-P plating layer sequentially

laminated between the nonmagnetic substrate and the magnetic recording layer.

The pretreatment layer achieves sufficient plating layer thickness, satisfactory adhesiveness with the substrate, and homogeneity. Therefore, a magnetic recording medium having a well-performed magnetic recording layer can be

5 obtained.

What is claimed is:

1. A method of pretreating a nonmagnetic substrate before forming a
5 magnetic recording layer thereon, the method comprising steps of:
 cleaning a surface of the substrate;
 forming a Ni layer on the surface of the nonmagnetic substrate after
 cleaning;
 forming a palladium layer that is a catalyst layer on the Ni layer; and
10 forming a Ni-P plating layer on the palladium layer.
2. The method according to claim 1, wherein
 the substrate is a glass substrate;
 the step of cleaning the substrate surface includes a step of degreasing
15 the substrate surface and a step of activating the substrate surface;
 the step of forming the Ni layer includes a step of applying a nickel
 chelate agent or a nickel soap agent, a step of metallizing baking the
 nickel chelate agent or the nickel soap agent, and a step of activating the
 metallized Ni layer;
20 the step of forming the palladium layer includes a step of converting
 the palladium layer to a catalyst layer; and
 the step of forming the Ni-P plating layer includes a step of electroless
 plating the Ni-P plating layer and a following step of heating treatment.
- 25 3. The method according to claim 1 or 2, wherein

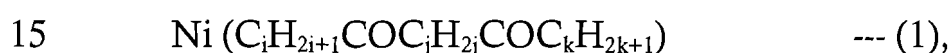
the step of forming the Ni layer includes a step of baking a nickel chelate agent or a nickel soap agent to form a nickel metallic film;

the step of forming the palladium layer includes a step of bonding palladium with a surface of the nickel metallic film; and

5 the step of forming the Ni-P plating layer is carried out by electroless plating after the step of bonding, and includes a subsequent step of heating treatment.

4. The method according to claim 1, wherein surface roughness of the
10 substrate surface after the pretreatment is at most 0.5 nm and micro surface waviness is at most 0.5 nm.

5. The method according to claim 2, wherein the nickel chelate agent has a structure represented by formula (1):



wherein i, j, and k are positive integers.

6. The method according to claim 2, wherein the nickel soap agent has a structure represented by one of $\text{Ni}(-\text{OOCCH}(\text{C}_n\text{H}_{2n+1})\text{C}_p\text{H}_{2p+1})_2$ and
20 $\text{Ni}(-\text{OOC C}_m\text{H}_{2m+1})_2$

wherein m, n, and p each are a positive integer of one or greater.

7. The method according to claim 2, wherein the step of metallizing baking is carried out in an inert gas atmosphere and at a temperature in a
25 range of 250°C to 400°C.

8. The method according to claim 2, wherein the step of converting the palladium layer to a catalyst layer is a treatment of palladium chloride.

5 9. The method according to claim 1 or 2, wherein the Ni-P plating layer contains phosphorus in a range of 1.0 wt% to 13.0 wt% and has a thickness at least 1.0 μm .

10 10. The method according to claim 2, wherein the heating treatment is carried out at a temperature in a range of 250°C to 300°C for at least 1 hr.

11. A magnetic recording medium comprising a nonmagnetic substrate, a magnetic recording layer formed above the substrate, and a pretreatment layer formed between the substrate and the magnetic recording layer by the
15 method of pretreating the substrate defined by any one of claims 1 through 10, the pretreatment layer comprising a nickel adhesion layer formed on the substrate, a palladium catalyst layer formed on the adhesion layer, and a Ni-P plating layer formed on the catalyst layer.