

Perpendicular Magnetic Recording Medium And A Method For Manufacturing the Same

Technical field of the invention

- 5 The present invention relates to a perpendicular magnetic recording medium mounted on various magnetic recording apparatuses, and to a manufacturing method therefor.

Background of the invention

- 10 A perpendicular magnetic recording system in which a direction of recording magnetization is perpendicular to a medium surface is drawing attention as a technology for achieving high density in magnetic recording to substitute for a conventional longitudinal magnetic recording system.
- 15 A perpendicular magnetic recording medium is mainly composed of a magnetic recording layer of hard magnetic material, an underlayer for aligning the magnetic recording layer to an aimed orientation, a protective layer for protecting a surface of the magnetic recording layer, and a backlining layer of soft magnetic material. The backlining layer concentrates magnetic flux
- 20 generated by a magnetic head used for recording on the recording layer. The soft magnetic backlining layer, which can enhance performance of a medium, can be omitted since recording is possible without a backlining layer.

To achieve high recording density in a perpendicular magnetic recording

medium, low noise compatible with high thermal stability is essential as in a longitudinal magnetic recording medium.

In the field of longitudinal magnetic recording media, various compositions
5 and structures of the magnetic recording layer and materials for the nonmagnetic underlayer have been proposed so far. Practically used magnetic recording layers employ an alloy composed mainly of cobalt and chromium (hereinafter referred to as a CoCr alloy). The chromium segregates to a grain boundary region and becomes a principal component of the grain boundary region to
10 obtain magnetically isolated magnetic grains. Another magnetic recording layer is proposed in which principal component of the grain boundary region has been oxide or nitride. (See US Patent No. 5,679,473 and Japanese Unexamined Patent Application Publication No. 2001-101651)

15 The above-mentioned magnetic materials in longitudinal magnetic recording media can also be applied to perpendicular magnetic recording media by, for example, providing an underlayer that controls crystal orientation to achieve perpendicular magnetic anisotropy. Reduction of magnetic interaction between magnetic grains is a problem to attain high recording density in
20 perpendicular magnetic recording media, too.

An example using a CoCr alloy is disclosed in Japanese Unexamined Patent Application Publication No. 2002-358615. The magnetic recording layer in the reference uses CoCrPt-X, and the chromium concentration is made higher

in the grain boundary region than in the crystalline grains, obtaining a segregation structure.

5 Examples using oxide or nitride as the principal component in the grain boundary region are disclosed in Japanese Unexamined Patent Application Publication No. 2000-306228 and Japanese Unexamined Patent Application Publication No. 2000-311329. A segregation structure is obtained by heat treatment in the references.

10 Problems to be solved by the invention

A magnetic recording layer comprising grain boundary region principally composed of oxide or nitride can drastically reduces media noise in comparison with a magnetic recording layer that uses a conventional CoCr alloy with a principal component of chromium in the grain boundary region, as disclosed in
15 T. Oikawa et al., IEEE Trans. Magn., vol. 38, No. 5 (Sept. 2002), pp. 1976-1978, “Microstructure and Magnetic Properties of CoPtCr-SiO₂ Perpendicular Recording Media”.

Perpendicular magnetic recording media are recently proposed in which a
20 magnetic recording layer is constructed with a multiple of function-separated layers to achieve not only reduction of media noise but also high thermal stability simultaneously.

Japanese Unexamined Patent Application Publication No. 2002-230735

discloses a multi-layered structure of CoCrPt alloy layers containing chromium and platinum with concentrations different from layer to layer. Japanese Unexamined Patent Application Publication No. 2003-168207 discloses a magnetic recording layer consisting of lower and upper two magnetic layers.

- 5 The lower magnetic layer comprises grain boundary region principally composed of oxide or nitride, and the upper magnetic layer includes a CoCr alloy for the purpose of enhancement of corrosion resistance.

To attain higher recording density in a perpendicular magnetic recording
10 medium, further reduction of media noise is essential. To reduce the media noise, a structure of a magnetic recording layer disclosed in Japanese Unexamined Patent Application Publication No. 2003-168207 is appropriate, in which a magnetic recording layer consists of upper and lower two magnetic layers and grain boundary region of the lower magnetic layer is principally
15 composed of oxide or nitride.

To achieve high density by virtue of reduction in media noise and simultaneously high reliability by virtue of excellent corrosion resistance, a structure in Japanese Unexamined Patent Application Publication No. 2003-
20 168207 may be appropriate in which a CoCr alloy is included in the upper magnetic layer. The Japanese Unexamined Patent Application Publication No. 2003-168207 further discloses post heating that is conducted after depositing lower and upper two magnetic layers composing a magnetic recording layer, to improve electromagnetic conversion characteristic as well as the corrosion

resistance.

For further improvement of the electromagnetic conversion characteristic in the structure of Japanese Unexamined Patent Application Publication No. 5 2003-168207, magnetic interaction needs to be decreased in the CoCr alloy layer of the upper magnetic layer composing the magnetic recording layer. To reduce the magnetic interaction, segregation of chromium to grain boundary region is desirably more promoted. However, the post heating as disclosed in Japanese Unexamined Patent Application Publication No. 2003-168207 fails to solve this 10 problem.

To improve electromagnetic conversion characteristic, a structure might be conceived that comprises a first magnetic layer of the magnetic recording layer of CoCrPt-SiO₂ disclosed in T. Oikawa et al., IEEE Trans. Magn., vol. 38, No. 5 15 (Sept. 2002), pp. 1976-1978, "Microstructure and Magnetic Properties of CoPtCr-SiO₂ Perpendicular Recording Media" and a second magnetic layer of the CoCrPt alloy film used for the construction in Japanese Unexamined Patent Application Publication No. 2002-230735. However, no concrete construction in such type of structure has ever been disclosed.

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Thus, a perpendicular magnetic recording medium is never disclosed that satisfies high recording density by virtue of improvement in electromagnetic conversion characteristic and high reliability by virtue of enhancement of corrosion resistance simultaneously.

Therefore, an object of the present invention is to provide a perpendicular magnetic recording medium and a method therefore that achieve both high recording density and high reliability in a medium comprising a magnetic recording layer consisting of a plurality of magnetic layers.

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Means to solve the problem

A perpendicular magnetic recording medium according to the present invention comprises a nonmagnetic substrate; an underlayer laminated over the substrate; a magnetic recording layer that consists of a lower magnetic layer
10 formed on the underlayer, the lower magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region containing oxide or nitride as a principal component, and an upper magnetic layer formed on the lower magnetic layer, the upper magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region in which concentration of non-ferromagnetic component
15 is higher than the non-ferromagnetic component in the ferromagnetic grains; and a covering layer formed on the upper magnetic layer; wherein the upper magnetic layer is formed after a heat treatment is conducted to the substrate having the lower magnetic layer formed.

20 The ferromagnetic grains in the lower magnetic layer contain cobalt as a principal component, and preferably contain platinum.

Advantageously, the nonmagnetic grain boundary region in the lower magnetic layer is substantially composed of oxide or nitride of at least an

element selected from Cr, Al, Ti, Si, Ta, Hf, Zr, Y, and Ce.

Advantageously, the ferromagnetic grains in the upper magnetic layer contain cobalt as a principal component, and at least chromium.

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Advantageously, the nonmagnetic grain boundary region in each of the upper magnetic layer contains chromium as one of the non-ferromagnetic components and concentration of the chromium is higher than concentration of the chromium in the ferromagnetic grains.

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Advantageously, the lower magnetic layer includes ferromagnetic grains composed of a CoPtCr alloy and nonmagnetic grain boundary region containing SiO₂; and the upper magnetic layer includes ferromagnetic grains composed of a CoPtCrB alloy and nonmagnetic grain boundary region in which concentration
15 of the chromium is higher than the chromium in the ferromagnetic grains.

Advantageously, a seed layer is further provided beneath the underlayer.

The seed layer can be composed of a NiFe alloy or a NiFe alloy containing
20 at least an additive element selected from B, Si, Nb, and Mo.

The seed layer can also be composed of cobalt or a cobalt alloy containing at least an additive element selected from B, Si, Nb, Mo, Fe, and Ni.

Advantageously, a soft magnetic backlining layer is further provided between the substrate and the underlayer or between the substrate and the seed layer,

5 Advantageously, the upper magnetic layer has approximately same grain size of the ferromagnetic grains and same width of the grain boundary region as those of the lower magnetic layer.

An aspect of the present invention is a method for manufacturing a
10 perpendicular magnetic recording medium. The method comprises a step of preparing a nonmagnetic substrate, a step of depositing an underlayer over the substrate, a process of depositing the magnetic recording layer on the underlayer, and a step of depositing a covering layer on the magnetic recording layer. The process of forming the magnetic recording layer comprises a step of depositing a
15 lower magnetic layer on the underlayer, the lower magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region composed of oxide or nitride, a step of conducting a heat treatment to the substrate having the lower magnetic layer formed, and a step of depositing an upper magnetic layer on the lower magnetic layer, the upper magnetic layer including ferromagnetic
20 grains and nonmagnetic grain boundary region in which concentration of non-ferromagnetic component is higher than the non-ferromagnetic component in the ferromagnetic grains.

By conducting the intermediate heat treatment, the upper magnetic layer

grows retaining approximately same grain size of the ferromagnetic grains and same width of the grain boundary region as those of the lower magnetic layer.

Brief description of drawings

- 5 Fig. 1 is a sectional view of a structure of a perpendicular magnetic recording medium according to first aspect of embodiment of the present invention.

Fig. 2 illustrates a surrounding structure of a lower magnetic layer constructing a magnetic recording layer.

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Fig. 3 illustrates a surrounding structure of an upper magnetic layer constructing a magnetic recording layer.

- Fig. 4 is a flow chart showing a manufacturing method for a perpendicular
15 magnetic recording medium according to second aspect of embodiment of the present invention.

Fig. 5 is a sectional view showing a structure of a magnetic recording layer formed by a manufacturing method including an intermediate heating step.

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Fig. 6 is a sectional view showing a structure of a magnetic recording layer formed by a manufacturing method without an intermediate heating step.

Best mode for embodiment of the invention

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

5 First aspect of embodiment of the invention

First aspect of embodiment will be described with reference to the Figs. 1, 2, and 3 in the following.

Fig. 1 shows a cross section of an exemplary structure of a perpendicular
10 magnetic recording medium according to the invention.

Referring to Fig. 1, a perpendicular magnetic recording medium 1
comprises a substrate 11 and layers sequentially laminated on the substrate, the
layers including an underlayer 14, a magnetic recording layer 15 that is a
15 lamination of a lower magnetic layer 151 and an upper magnetic layer 152, a
protective layer 16, and a liquid lubricant layer 17.

A nonmagnetic substrate 11 can be made of a Ni-P plated aluminum alloy,
strengthened glass, or crystallized glass, each being used in a conventional
20 magnetic recording medium.

The underlayer 14 is preferably composed of a metal or alloy having a
hexagonal closest packed (hcp) structure or a metal or alloy having a face
centered cubic (fcc) structure.

The preferable metals with the hcp structure includes Ti, Zr, Ru, Zn, Tc, and Re. The preferable metals with the fcc structure include Cu, Rh, Pd, Ag, Ir, Pt, Au, Ni, and Co.

5 Ru, Ir, Pt, Au, and alloys containing these metals, in particular, are used by preference, because these metals and alloys scarcely react with oxygen or nitrogen contained in nonmagnetic grain boundary region of a lower magnetic layer 151 that is described later, thus, are improbable to disturb crystal orientation of cobalt that is a principal component of ferromagnetic grains in the
10 lower magnetic layer 151. Thickness of the underlayer is desired to be a minimum value, while a thickness of 3 nm or more is preferable to ensure crystal growth of the underlayer itself.

Protective layer 16 can be a thin film principally composed of carbon.
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Liquid lubricant layer 17 can be composed of perfluoropolyether lubricant.

Advantageously, a seed layer 13 and a soft magnetic backlining layer 12 are further provided as described below.

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A seed layer 13 is provided beneath the underlayer 14 to improve orientation of the underlayer.

The seed layer can be composed of a nonmagnetic material or a soft

magnetic material. When a soft magnetic backlining layer 12 is formed beneath the seed layer, a soft magnetic material is preferable because the material functions partly as a soft magnetic backlining layer.

- 5 The soft magnetic material for the seed layer 13 can be selected from nickel-based alloys such as NiFe, NiFeNb, NiFeB, and NiFeCr, and from cobalt and cobalt-based alloys such as CoB, CoSi, CoNi, and CoFe. These materials for the seed layer take preferably fcc or hcp structure as in the underlayer 14. The seed layer may consist of plural layers of the above materials, each layer
10 functioning separately to ensure crystalline lattice consistency or to control grain size.

A soft magnetic backlining layer 12 is preferably provided for controlling magnetic flux generated by a magnetic head and assist magnetic recording.

- 15 When a seed layer 13 is provided under an underlayer 14, the soft magnetic backlining layer 12 is provided under the seed layer 13.

- 20 The soft magnetic backlining layer 12 can be made of a material selected from a crystalline alloy such as a NiFe alloy, a Sendust (FeSiAl) alloy, or a CoFe alloy, a fine crystalline material such as FeTaC, CoTaZr, CoFeNi, and CoNiP, and an amorphous cobalt alloy such as CoNbZr. An optimum thickness of the backlining layer 12 varies depending on a structure and characteristics of a magnetic head used for magnetic recording. When the soft magnetic backlining layer is formed in sequential depositions with other layers, the

preferable thickness is in a range of 10 nm to 500 nm considering productivity. In the case of preliminary deposition by plating on a nonmagnetic substrate before deposition of other layers, the thickness may be as thick as several microns.

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Magnetic recording layer

Now, a magnetic recording layer 15 is described here in detail.

A magnetic recording layer 15 is a lamination of plural magnetic layers of
10 different materials: a lower magnetic layer 151 and an upper magnetic layer 152.

The lower magnetic layer 151 shown in Fig. 2 has a granular structure in which ferromagnetic grains 20 are surrounded by nonmagnetic grain boundary region 21 of principal component of oxide or nitride.

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The ferromagnetic grains 20 in the lower magnetic layer 151 are preferably composed of a principal component of cobalt and at least platinum.

The ferromagnetic grains 20 favorably further contain at least an element
20 selected from Ta, B, Cr, Nb, Ag, Mo, W, Pd, and Cu for the purpose of grain size control and crystallinity improvement. Appropriate materials for the nonmagnetic grain boundary 21 of the lower magnetic layer 151 includes oxides or nitrides of at least one element selected from Cr, Al, Ti, Si, Ta, Hf, Zr, Y, and Ce.

An upper magnetic layer 152 shown in Fig. 3 has a structure in which ferromagnetic grains 30 are surrounded by nonmagnetic grain boundary region 31 principally composed of chromium.

- 5 The ferromagnetic grains 30 in the upper magnetic layer 152 are preferably composed of a principal component of cobalt, and at least chromium.

 The ferromagnetic grains 30 favorably further contain at least one element selected from Pt, Ta, B, Nb, Ag, Mo, W, Pd, and Cu, in addition to the cobalt
10 and chromium.

 The nonmagnetic grain boundary region 31 in the upper magnetic layer 152 contains a non-ferromagnetic component preferably in a concentration higher than a concentration of the same non-ferromagnetic component in the
15 ferromagnetic grains 30. In particular, the nonmagnetic grain boundary region 31 contains preferably a non-ferromagnetic component of chromium and the chromium concentration is higher than a chromium concentration in the ferromagnetic grains. This condition is called as chromium-rich.

- 20 The magnetic recording layer 15 having the above-described structure is subjected to heat treatment of the substrate after deposition of the lower magnetic layer 151 and before deposition of the upper magnetic layer 152. Preferable conditions of heat treatment of the magnetic recording layer are a temperature higher than 150°C that produces sufficient effect and a time

duration in the range of 1 sec to 10 min considering productivity.

Second aspect of embodiment of the invention

Second aspect of embodiment of the present invention is described below
5 referring to Figs. 4 through 6 and Table 1. The same parts as in the first aspect
of embodiment are given the same symbols and are not described repeatedly.

A manufacturing method for a perpendicular magnetic recording medium 1
described in the first aspect of embodiment is described here as a second aspect
10 of invention.

Manufacturing method

Fig. 4 is a flow chart showing an example of manufacturing method of
perpendicular magnetic recording media. In step S1, layers up to an underlayer
15 14 are sequentially formed on a nonmagnetic substrate 11.

A chemically reinforced glass substrate having a smooth surface (for
example, N-5 glass substrate manufactured by HOYA Corporation) is used for
the nonmagnetic substrate 11. After cleaning, the substrate is put into a
20 sputtering apparatus. A soft magnetic backlining layer 12 of CoZrNb 150 nm
thick is formed using a target of $\text{Co}_{88}\text{Zr}_4\text{Nb}_8$ under argon gas pressure of 5
mTorr on the substrate. Subsequently, a seed layer 13 of NiFeSiB 10 nm thick
is formed using a target of a soft magnetic nickel-based alloy $\text{Ni}_{84}\text{Fe}_{12}\text{Si}_3\text{B}_1$
under argon gas pressure of 20 mTorr.

On the seed layer 13, a ruthenium underlayer 14 with a thickness of 10 nm is formed using a ruthenium target under argon gas pressure of 30 mTorr.

5 In step S2, a lower magnetic layer 151 consisting a part of a magnetic recording layer 15 is formed. A lower magnetic layer 151 of CoPtCr-SiO₂ shown in Fig. 2 having thickness of 8 nm is formed by means of an RF sputtering method using a target of (Co₇₂Pt₁₈Cr₁₀)₉₃(SiO₂)₇ under argon gas pressure of 30 mTorr.

10 In step S3, the nonmagnetic substrate 11 having the lower magnetic layer 151 of CoPtCr-SiO₂ formed thereon is subjected to heat treatment, which is referred to as intermediate heating. The intermediate heating is conducted with a lamp heater in a heating chamber at a substrate temperature of 200°C for 10 sec.

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In step S4, after the intermediate heating, an upper magnetic layer 152 consisting a part of the magnetic recording layer 15 is formed.

20 An upper magnetic layer 152 of CoCrPtB as shown in Fig. 3 having a thickness of 6 nm is formed, on the heat-treated lower magnetic layer 151 of CoPtCr-SiO₂, using a target of Co₆₆Cr₂₀Pt₁₀B₄ under argon gas pressure of 5 mTorr.

In step S5, layers from a protective layer 16 and above are sequentially

formed as a covering layer.

A carbon protective layer 16 with a thickness of 3 nm is deposited by an ion beam deposition method. After that, the substrate is taken out from the vacuum chamber.

Then, a liquid lubricant layer 17 of perfluoropolyether 2 nm thick is formed by dip-coating method. Thus, a perpendicular magnetic recording medium 1 is produced.

10

The deposition steps of the above described layers were conducted by a DC magnetron sputtering method except for deposition steps of the lower magnetic layer 151 and of the protective layer 16.

15 Intermediate heating of a magnetic recording layer

The following describes benefits of the intermediate heating of a magnetic recording layer consisted of the lower and upper magnetic layers 151 and 152.

Distinctive features in structure produced by the intermediate heating

20 (1) Fig. 5 shows a sectional structure of a magnetic recording layer 15 of Example of embodiment according to the invention. Intermediate heating is carried out in the Example of embodiment of the invention. Fig. 6 shows a sectional structure of a magnetic recording layer 15 in the case without intermediate heating for comparison.

Positional relationship between the upper and lower layers of the magnetic recording layer 15 in Fig. 5 is basically such that every crystalline grain in the upper magnetic layer 152 positions on a crystalline grain in the lower magnetic layer 151, and every grain boundary in the upper magnetic layer 152 positions on grain boundary in the lower magnetic layer 151.

In a magnetic layer with intermediate heating as shown in Fig. 5, the upper magnetic layer 152 grows directly reflecting the grain size and the grain boundary width in the lower magnetic layer 151.

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In contrast, in the case without intermediate heating as shown in Fig. 6, while the upper magnetic layer 152 grows reflecting grain size and grain boundary width in the vicinity of interface with the lower magnetic layer 151, as the growth proceeds, the grain boundary width narrows and the grain size enlarges.

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The ferromagnetic crystalline grain A in Fig. 6, for example, swells corresponding to shrinkage of the surrounding grain boundary. The ferromagnetic grain B in Fig. 6 enlarges (doubles) joining with a neighboring grain together.

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Thus, in the case without intermediate heating, the mean grain size increases and the mean grain boundary width decreases.

(2) In a magnetic recording layer 15 of the example as shown in Fig. 5, a mean grain boundary width is in a range of 0.5 to 2.0 nm and a mean grain size is in a range of 4 to 12 nm.

5 The grain size and crystallinity can be determined depending on composition of the magnetic recording layer 15, additives to the magnetic recording layer 15, deposition process of the magnetic recording layer 15, and grain size and grain boundary structure of an underlayer 14.

10 In the upper magnetic layer 152 without intermediate heating as shown in Fig. 6, mean grain boundary width is smaller and mean grain size is larger than those described above for the case with intermediate heating. In a specific example with intermediate heating, mean grain boundary width = 1.1 nm and mean grain size = 7.3 nm, while in an example without intermediate heating,
15 mean grain boundary width = 0.7 nm and mean grain size = 9.5 nm.

In the magnetic recording layer with the structure shown in Fig. 5, control of grain size and crystallinity can be carried out by selecting additive element to the ferromagnetic crystalline grains, as described below.

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In the lower magnetic layer 151, grain size can be made fine and intergranular magnetic interaction can be reduced by adding at least an element selected from Ta, B, Nb, Mo, W, and Cr.

In the lower magnetic layer 151, crystallinity can be enhanced and crystalline magnetic anisotropy constant K_u can be elevated by adding at least an element selected from Ag, Pd, and Cu.

- 5 In the upper magnetic layer 152, grain size can be made fine and intergranular magnetic interaction can be reduced by adding at least an element selected from Ta, B, Nb, Mo, and W.

- 10 In the upper magnetic layer 152, crystallinity can be enhanced and magnetic anisotropy constant K_u can be elevated by adding at least an element selected from Ag, Pd, Cu, and Pt.

Effects of the intermediate heating

- (1) An upper magnetic layer 152 in the structure of a magnetic recording layer
15 15 shown in Fig. 5 has a benefit that the upper magnetic layer grows retaining the same grain size and grain boundary width as in the lower magnetic layer 151 and facilitate favorable chromium segregation. As a result, SNR enhances according to the following reasoning.

- 20 Since the same grain size and grain boundary width are maintained, the magnetic grains physically separate each other, so that intergranular magnetic interaction reduces, which decreases magnetic cluster size (described later), which in turn, decreases width of a transition region between bits, which means reduction in bit transition noise.

Because of favorable chromium segregation, [1] cobalt proportion in ferromagnetic grains increases, which increases magnetic flux density in each crystalline grain, which increases output signal. The favorable chromium segregation, on the other hand, causes [2] small cobalt proportion in
5 nonmagnetic grain boundary region, which reduces magnetic interaction, which in turn, decreases magnetic cluster size, which reduces bit transition noise.

In the magnetic recording layer in the structure shown in Fig. 5, if every grain independently makes inversion of magnetization, it is an ideal state in
10 which no magnetic interaction exists between the grains. In actual, however, magnetic interaction acts between the grains. Consequently, several grains or more form a magnetization inversion unit. Such a group of the grains is called a magnetic cluster. Reduction of a size of the magnetic cluster reduces noises and raises SNR.

15

In contrast, a magnetic recording layer in the structure shown in Fig. 6, in a case in which intermediate heating is not conducted, but post heating after deposition of the upper magnetic layer 152 is conducted for example, as in the case of Comparative Example 4 in Table 1, grain sizes of the lower magnetic
20 layer 151 may be made uniform in some degree and chromium segregation in the upper magnetic layer 152 may occur. But, they can occur to a limited extent.

Because a course of growing process of the upper magnetic layer 152 is similar to the case with no heating, as Comparative Example 3 in Table 1,

distance between the grains is small and thus, a size of magnetic clusters increases, resulting in degradation in SNR.

As such, by conducting the intermediate heating as in the Examples of
5 embodiment of the invention, a magnetic cluster size as a whole in the magnetic recording layer 15 is decreased; this enables low noise and improved SNR.

(2) The benefits of the intermediate heating described above are summarized in the following.

- 10 a) By subjecting the lower magnetic layer 151 to intermediate heating, deviation in grain sizes is decreased and the grain sizes are made uniform. In this stage, mean grain size and mean grain boundary width remain almost unchanged.
- 15 b) If an upper magnetic layer 152 is formed on the lower magnetic layer 151 without heating, the grain size increases and the grain boundary width decreases in the upper magnetic layer with increase of film thickness of the upper magnetic layer.

20 In contrast, when the upper magnetic layer 152 is formed after intermediate heating according to the invention, the increase in grain size and the decrease in grain boundary width are suppressed and the upper magnetic layer 152 grows following the grain size and the grain boundary width of the lower magnetic layer 151.

c) In an upper magnetic layer 152 formed after intermediate heating, chromium segregation to the nonmagnetic grain boundary region 31 is promoted as shown in Fig. 3 and the nonmagnetic grain boundary region 31 becomes more chromium-rich than the ferromagnetic crystalline grain 30. Consequently, a layer of a CoCr alloy, a CoCrPt alloy, or a CoCrPtB alloy is allowed to be employed for ferromagnetic grains of the upper magnetic layer 152.

d) A surface of an upper magnetic layer 152 after the intermediate heating exhibits better flatness than a post heated surface of a conventional magnetic recording layer.

(3) In summary, the intermediate heating:

- [1] makes grain sizes uniform,
- [2] suppresses increase in grain sizes in the upper magnetic layer 152,
- 15 [3] promotes chromium segregation in the upper magnetic layer 152,
- [4] allows employing a CoCr alloy, a CoCrPt alloy, or a CoCrPtB alloy in the upper magnetic layer 152, and
- [5] improves flatness of the surface of the upper magnetic layer 152.

20 The effects [1], [2], and [3] lead to enhancement of output signal and reduction of noises simultaneously, which means improvement in SNR of an evaluation of electromagnetic conversion characteristics, achieving high recording density.

Recording density is determined by characterizing BER (bit error rate). A high BER means read out at the recording density impossible. A threshold value of a BER depends on a signal processing circuit. For example, a certain signal processing circuit correctly reads out data at a $BER = 10^{-5}$ or less, while another
5 circuit only permits a $BER = 10^{-6}$ or less. The BER is generally improved with increase of SNR. Therefore, enhancement in SNR can be employed as an index for improvement of recording density in the present invention, too. For example, an enhancement in SNR by 1 dB is equivalent to improvement of BER of one to one- and- a- half orders of magnitude, and an enhancement in SNR by 2 dB is
10 equivalent to improvement of BER of two to three orders of magnitude.

The effects [4] and [5] indicated above increases coverage with the protective layer 16, which reduces cobalt precipitation by ICP characterization, which means high resistance to corrosion. Therefore, high reliability can be
15 achieved.

Evaluation of a perpendicular magnetic recording medium

Table 1 shows evaluation result of the example (Example of invention) manufactured by a method of the invention as well as evaluation results for
20 comparative examples.

Items of evaluation are conditions of thickness and heating, SNR, which is an evaluation of electromagnetic conversion characteristics, and ICP analysis of cobalt precipitation.

Table 1

	Structure of magnetic recording layer and heating process				Electro-magnetic characteristics	ICP evaluation
	Thickness of CoPtCr-SiO ₂ lower magnetic layer (nm)	Intermediate heating	Thickness of CoCrPtB upper magnetic layer (nm)	Post heating	SNR at 367 kFCI (dB)	Amount of cobalt precipitation (ng/cm ²)
Ex	8	Done	6	None	15.06	0.074
CEx 1	8	None	0	None	13.42	0.625
CEx 2	14	None	0	None	13.56	0.684
CEx 3	8	None	6	None	12.80	0.216
CEx 4	8	None	6	Done	13.93	0.213

Ex: Example of invention

CEx: Comparative Example

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In evaluation of the Example of embodiment of the invention, comparison was made with Comparative Examples 1 through 4 produced as follows.

Comparative Example 1

10 A perpendicular magnetic recording medium 1 of Comparative Example 1 was produced in the same manner as in the Example of invention except that heating after deposition of a lower magnetic layer of CoPtCr-SiO₂ was not conducted and the upper magnetic layer CoCrPtB as in Example of invention was not deposited.

15

Comparative Example 2

A perpendicular magnetic recording medium 1 of Comparative Example 2 was produced in the same manner as in Comparative Example 1 except that a thickness of the lower magnetic layer of CoPtCr-SiO₂ was 14 nm.

5

Comparative Example 3

A perpendicular magnetic recording medium 1 of Comparative Example 3 was produced in the same manner as in the Example of invention except that no heating was conducted after deposition of the lower magnetic layer of CoPtCr-SiO₂.

10

Comparative Example 4

A perpendicular magnetic recording medium 1 of Comparative Example 4 was produced in the same manner as in Example of invention except that no intermediate heating was conducted after deposition of the lower magnetic layer 151 of CoPtCr-SiO₂, and post heating was conducted after an upper magnetic layer of CoCrPtB was laminated.

15

Conditions for the post heating were 200°C and 10 sec, which were the same as the heating after deposition of the lower magnetic layer 151 of CoPtCr-SiO₂ and before deposition of the upper magnetic layer 152 of CoCrPtB conducted in Example of invention.

20

The thus produced five types of perpendicular magnetic recording media

were characterized by electromagnetic conversion characteristic in view of recording density and ICP analysis of amount of cobalt precipitation from the magnetic recording layer in view of corrosion resistance.

- 5 For evaluation of electromagnetic conversion characteristic, SNR was measured by a spinning stand tester equipped with a GMR head.

Measurement of amount of cobalt precipitation was carried out by placing a sample in a high temperature and humidity environment of a temperature of
10 85°C and relative humidity of 80 % for 96 hr, then shaking the sample in 50 ml of pure water for 3 min to extract the solved out cobalt, and measuring the concentration by means of ICP emission spectroscopic analysis.

Though Table 1 gives the SNR values at a linear recording density of 367
15 kFCI as an example, it has been confirmed that order of superiority does not change at other linear recording densities.

First, a comparison is made between Comparative Example 1 and Comparative Example 3. No heating was conducted in both Comparative
20 Examples 1 and 3. Comparative Example 1 resulted in SNR=13.42 dB and an amount of cobalt precipitation=0.625 ng/cm², and in Comparative Example 3, SNR=12.80 dB and an amount of cobalt precipitation=0.216 ng/cm².

Comparative Example 3 suppressed the amount of cobalt precipitation to about one third, though exhibiting lower SNR by 0.6 dB.

As this comparison indicates, cobalt precipitation is remarkably suppressed
5 by laminating the upper magnetic layer 152 of CoCrPtB that comprises the grain boundary region 31 mainly composed of chromium and precipitates less cobalt than the lower magnetic layer 151 of CoPtCr- SiO₂ that comprises the grain boundary region 21 mainly composed of an oxide SiO₂.

10 However, noises are greater and SNR are worse in Comparative Example 3 because intergranular magnetic interaction is relatively larger in the upper magnetic layer 152 of CoCrPtB.

Total thickness of the magnetic recording layers 15 in Comparative
15 Examples 1 and 3 are different: 8 nm in Comparative Example 1 and 14 nm in Comparative Example 3.

To confirm the difference in SNR and difference in cobalt precipitation is not simply due to an effect of difference in film thickness, Example 2 was
20 prepared having a thickness 14 nm of a lower magnetic layer 151 of CoPtCr-SiO₂.

The Comparative Example 2 resulted in SNR=13.56 dB and an amount of cobalt precipitation=0.684 ng/cm². No heating was conducted in Comparative

Example 2.

Comparing the Comparative Example 2 with Comparative Example 1, SNR improved by about 0.1 dB and cobalt precipitation increased by 0.06
5 ng/cm². Whereas, comparing with Comparative Example 3, cobalt precipitation increased to about 3 times as large value though SNR improved by 0.8 dB.

This tendency is equivalent to the comparison between Comparative Examples 1 and 3. Thus, the degradation of SNR and the improvement in cobalt
10 precipitation by laminating an upper magnetic layer 152 of CoCrPtB on the lower magnetic layer 151 of CoPtCr- SiO₂ are certainly not simply due to an effect of film thickness.

Next, a comparison is made between Comparative Examples 3 and 4.
15 Comparative Example 4 resulted in SNR=13.93 dB and an amount of cobalt precipitation=0.213 ng/cm². Post heating was conducted in the Comparative Example 4. Comparative Example 4, which was heated after deposition of the upper magnetic layer 152 of CoCrPtB, improved SNR by 1.1 dB, while difference in amount of cobalt precipitation is 0.003 ng/cm² and
20 substantially equivalent.

The improvement in SNR indicates that the post heating promotes chromium segregation to the grain boundary region in the CoCrPtB layer and reduces intergranular magnetic interaction. The substantially equivalent cobalt

precipitation indicates that the heating does not change quality of the protective layer 16 and thus, does not alter cobalt precipitation.

Finally, a comparison is made between the Example of invention and
5 Comparative Example 4. The Example of invention resulted in SNR=15.06 dB and an amount of cobalt precipitation=0.074 ng/cm². An intermediate heating was conducted in the Example of invention.

The Example of invention is subjected to intermediate heating between the
10 steps of depositing the lower magnetic layer 151 of CoPtCr-SiO₂ and depositing the upper magnetic layer 152 of CoCrPtB. By virtue of the intermediate heating, the Example of invention, comparing with Comparative Example 4, improved SNR by 1.1 dB and suppressed the cobalt precipitation to a very small value of 0.074 ng/cm², which is about one third of that in Comparative Example 4.

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This intermediate heating between the deposition steps of the lower magnetic layer 151 of CoPtCr- SiO₂ and the upper magnetic layer 152 of CoCrPtB is precedent heating to deposition of the upper magnetic layer of CoCrPtB. It has been demonstrated that the precedent heating more promotes
20 chromium segregation and at the same time, more suppresses cobalt precipitation than the post heating.

As described so far, a magnetic recording layer 15 of the Example of invention is a lamination consisting of lower and upper magnetic layers 151 and

152, wherein the lower magnetic layer 151 comprises ferromagnetic grains 20 surrounded by nonmagnetic grain boundary region 21 principally composed of oxide or nitride, and the upper magnetic layer 152 comprises ferromagnetic grains 30 surrounded by nonmagnetic grain boundary region 31 principally
5 composed of chromium. As a result, a perpendicular magnetic recording medium manufactured by a method comprising a process to heat the nonmagnetic substrate 11 with a deposited lower magnetic layer before a step of forming an upper magnetic layer according to the invention achieves higher SNR than a type of magnetic recording medium with post heating (as
10 Comparative Example 4). At the same time, a magnetic recording medium of the invention drastically suppresses cobalt precipitation from a magnetic recording layer 15 in comparison with a type of magnetic recording medium without post heating (as Comparative Example 1 through 3). Therefore, a perpendicular magnetic recording medium is provided that achieves high
15 recording density by virtue of improvement in electromagnetic conversion characteristic and simultaneously high reliability by virtue of improvement in corrosion resistance.

Advantageous Effect of the invention

20 To solve the above problem, a perpendicular magnetic recording medium of the invention comprises a magnetic recording layer that is a lamination of magnetic layers composed of different materials.

A lower magnetic layer comprises ferromagnetic crystalline grains surrounded by nonmagnetic grain boundary region composed mainly of oxide or nitride and

an upper magnetic layer comprises ferromagnetic crystalline grains surrounded by nonmagnetic grain boundary region composed mainly of chromium. A heating process, intermediate heating, is inserted after the process of depositing the lower magnetic layer and before the process of depositing the upper magnetic layer. By virtue of the intermediate heating process, chromium segregation in the upper magnetic layer is remarkably promoted in comparison with a post heating process conducted after complete deposition of both lower and upper magnetic layers. As a result, electromagnetic conversion characteristic is further improved and at the same time, corrosion resistance is also enhanced. Therefore, high recording density and high reliability are simultaneously achieved in a perpendicular magnetic recording medium of the invention.

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Claims

1. A perpendicular magnetic recording medium comprising:
 - a nonmagnetic substrate;
 - 5 an intermediate layer laminated over the substrate;
 - a magnetic recording layer comprising
 - a first magnetic layer formed on the intermediate layer, the first magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region containing oxide and/or nitride, and
 - 10 a second magnetic layer formed on the first magnetic layer, the second magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region lacking oxide and nitride; and
 - a protective layer formed on the second magnetic layer, wherein the mean grain size of the first and second magnetic layers is the same and
 - 15 the mean width of the grain boundary region in the first and second magnetic layers is the same.
2. The perpendicular magnetic recording medium according to claim 1, wherein the ferromagnetic grains in the first magnetic layer contain cobalt
 - 20 as a principal component, and platinum or chromium, and the nonmagnetic grain boundary region in the first magnetic layer is substantially composed of oxide and/or nitride of at least an element selected from Cr, Co, Al, Ti, Si, Ta, Hf, Zr, Y and Ce.
- 25 3. The perpendicular magnetic recording medium according to claim

2, wherein the ferromagnetic grains in the first magnetic layer further contain at least an element selected from Ni, Ta, B, Nb, Ag, Mo, W, Pd, and Cu.

4. The perpendicular magnetic recording medium according to claim 1, wherein a concentration of each of non-ferromagnetic components in the nonmagnetic grain boundary region in the second magnetic layer is higher than a concentration of the same non-ferromagnetic component in the ferromagnetic grains in the second magnetic layer.

5. The perpendicular magnetic recording medium according to claim 1, wherein the ferromagnetic grains in the second magnetic layer contain a CoCr alloy as a principal component, and at least an element selected from Pt, Ta, B, Nb, Ag, Mo, W, Pd, and Cu; and the nonmagnetic grain boundary region contains at least chromium in a concentration that is higher than a concentration of chromium contained in the ferromagnetic grains in the second magnetic layer.

6. The perpendicular magnetic recording medium according to claim 1, wherein the intermediate layer is composed of an element having a hexagonal closest packed (hcp) structure selected from Ti, Re, Ru, Os, Zr, Zn, and Tc, or an alloy having the hcp structure containing at least an element selected from Ti, Re, Ru, Os, Zr, Zn, and Tc.

7. The perpendicular magnetic recording medium according to claim

1, wherein the intermediate layer is composed of an element having a face centered cubic (fcc) structure selected from Cu, Rh, Pd, Ag, Ir, Pt, Au, Ni, and Co, or an alloy having the fcc structure containing at least an element selected from Cu, Rh, Pd, Ag, Ir, Pt, Au, Ni, and Co.

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8. The perpendicular magnetic recording medium according to claim 1 further comprising a seed layer beneath the intermediate layer.

9. The perpendicular magnetic recording medium according to claim

10 8, wherein the seed layer is composed of either (i) a NiFe alloy or (ii) a NiFe alloy containing at least an additive element selected from B, Si, Nb, and Mo.

10. The perpendicular magnetic recording medium according to claim

15 8, wherein the seed layer is composed of either (i) cobalt or (ii) a cobalt alloy containing at least an additive element selected from B, Si, Nb, Mo, Fe, and Ni.

11. The perpendicular magnetic recording medium according to claim 8,

20 further comprising a soft magnetic backlining layer between the substrate and the seed layer.

12. The perpendicular magnetic recording medium according to claim 11, further comprising an underlayer deposited on the substrate and a magnetic

25 domain control layer deposited on the underlayer.

13. The perpendicular magnetic recording medium according to claim 1, further comprising a soft magnetic backlining layer between the substrate and the intermediate layer.

5 14. The perpendicular magnetic recording medium according to claim 13, further comprising an underlayer deposited on the substrate and a magnetic domain control layer deposited on the underlayer.

15. The perpendicular magnetic recording medium according to claim 10 1, wherein the second magnetic layer is formed after a heat treatment of the substrate having the first magnetic layer formed thereon.

16. A method for manufacturing a perpendicular magnetic recording medium comprising:

15 preparing a nonmagnetic substrate without preheating process;
depositing an intermediate layer on the substrate;
depositing a first magnetic layer on the intermediate layer by a sputtering method, the first magnetic layer comprising ferromagnetic grains and nonmagnetic grain boundary region including oxide and/or nitride
20 depositing a second magnetic layer on the first magnetic layer by a sputtering method, the second magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region lacking oxide and nitride, wherein the mean grain size of the first and second magnetic layers is the same and the mean width of the grain boundary region in the first and
25 second magnetic layers is the same; and

depositing a protective layer on the second magnetic layer; wherein a gas pressure during deposition of the first magnetic layer is 10 mTorr or more, and a gas pressure during deposition of the second magnetic layer is 15 mTorr or less.

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17. The method for manufacturing a perpendicular magnetic recording medium according to claim 16, further comprising heating the substrate after depositing the second magnetic layer in a deposition apparatus.

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18. The method for manufacturing a perpendicular magnetic recording medium according to claim 17, further comprising fast cooling the substrate in the deposition apparatus after heating.

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19. The method for manufacturing a perpendicular magnetic recording medium according to claim 16, further comprising depositing a seed layer on the substrate without heating process before depositing the intermediate layer.

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20. The method for manufacturing a perpendicular magnetic recording medium according to claim 16, further comprising depositing an underlayer on the substrate, depositing a magnetic domain control layer on the underlayer, and depositing a soft magnetic backlining layer on the magnetic domain control layer.

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21. A method for manufacturing a perpendicular magnetic medium comprising:

preparing a nonmagnetic substrate without preheating process;

depositing an intermediate layer on the substrate;

5 depositing a first magnetic layer on the intermediate layer by a sputtering method, the first magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region containing oxide and/or nitride;

heating the substrate having the first magnetic layer formed thereon;

10 depositing a second magnetic layer on the first magnetic layer by a sputtering method, the second magnetic layer including ferromagnetic grains and nonmagnetic grain boundary region lacking oxide and nitride, wherein the mean grain size of the first and second magnetic layers is the same and the mean width of the grain boundary region in the first and second magnetic layers is the same; and

15 depositing a protective layer on the second magnetic layer.

22. The method for manufacturing a perpendicular magnetic recording medium according to claim 21, further comprising depositing a seed layer right before depositing the intermediate layer.

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23. The method for manufacturing a perpendicular magnetic recording medium according to claim 21, further comprising depositing an underlayer on the substrate, depositing a magnetic domain control layer on the underlayer, and depositing a soft magnetic backlining layer on the

25 magnetic domain control layer.