

DESCRIPTION
FE-CO ALLOY SPUTTERING TARGET MATERIAL FOR
FORMING SOFT MAGNETIC FILM

TECHNICAL FIELD

[0001]

The present invention relates to an Fe-Co based alloy sputtering target material for forming a soft magnetic film.

5 BACKGROUND ART

[0002]

Recent progress in magnetic recording technology is noticeable, and the recording density of magnetic recording media becomes increased for larger drive capacity. However, in the case of the magnetic recording media for the longitudinal magnetic recording method that is now widely and commonly used, in order to achieve increased recording density, recording bits have to be miniaturized, and the high coercivity which can not be recorded by a recording head is required. Accordingly, a perpendicular magnetic recording method is investigated as a means for solving these problems and improving the recording density.

The perpendicular magnetic recording method is a method in which a magnetic film made of a perpendicular magnetic recording medium is formed such that the axis of easy magnetization is oriented in a perpendicular direction with respect to the medium plane, and which method is suitable for increased recording density since even if the recording density is increased, the demagnetizing field in bits remains small and the recording and reproducing properties deteriorate little. For the perpendicular magnetic recording method, a recording medium having a magnetic recording film layer increased in recording sensitivity and a soft magnetic film layer is developed.

[0003]

As a soft magnetic film made of this kind of magnetic recording medium, an amorphous soft magnetic alloy is adopted since excellent soft magnetic properties are required. As typical amorphous alloy for a soft magnetic film, an alloy film containing Fe, Co or Fe-Co alloy with additive elements, a Co-Zr-Nb alloy film, a Co-Zr-Ta alloy film and the like are already put to practical use (see PATENT DOCUMENT 1, for example).

DISCLOSURE OF THE INVENTION

TECHNICAL PROBLEM

[0004]

The soft magnetic film of a magnetic recording medium is required to have a high saturation flux density, and an Fe-Co alloy composed mainly of Fe having a high saturation flux density is preferably utilized. Further, among the Fe-Co alloys, an Fe-rich Fe-Co alloy is utilized when higher saturation flux is required.

Since the soft magnetic film of a magnetic recording medium is required to become an amorphous film, it is necessary to add an element which facilitates amorphization to the above described Fe-Co alloy, and B and Zr are adopted generally. However, when used for the soft magnetic film of a magnetic recording medium, the corrosion resistance is not enough only by these elements. Accordingly, some documents propose further addition of elements such as Al and Cr for improved corrosion resistance (see JP-A-2007-172783, JP-A-2007-284741, for example). However, there is the problem that the component composition of the soft magnetic film becomes complicated and therefore makes it difficult to control the composition and the components when making a sputtering target material.

[0005]

An object of the present invention is to provide an Fe-Co based alloy sputtering target material for forming a soft magnetic film that is used for a perpendicular magnetic recording medium or the like and solves the above described problem, the material having Fe-Co based alloy composition achieving amorphization and improvement in corrosion resistance possibly by addition of a single element.

TECHNICAL SOLUTION

[0006]

The present inventors have made various investigations on Fe-Co based alloy sputtering target materials for forming a soft magnetic film used for a perpendicular magnetic recording medium or the like and on additive elements to the Fe-Co alloy, and consequently found selecting Nb or Ta, and a suitable addition range thereof to attain the present invention.

[0007]

That is, the present invention provides an Fe-Co based alloy sputtering target material for forming a soft magnetic film, comprising: an Fe-Co alloy represented by the composition formula regarding an atomic ratio: $\text{Fe}_X\text{-Co}_{100-X}$, wherein $10 \leq X \leq 80$; and 10-20 atom% of one or two elements selected from Ta and Nb.

Preferably, the present invention provides an Fe-Co based alloy sputtering target material for forming a soft magnetic film, comprising 10-15 atom% of one or two elements selected from Nb and Ta.

Also preferably, the present invention provides an Fe-Co based alloy sputtering target material for forming a soft magnetic film of which the maximum magnetic permeability is 250 or less, or of which the PTF is 10% or more.

ADVANTAGEOUS EFFECTS

[0008]

The present invention can provide an Fe-Co based alloy sputtering target for forming a soft magnetic film of a perpendicular magnetic recording medium by which stable magnetron sputtering can be performed, resulting in a highly effective technology in producing a perpendicular magnetic recording medium.

BEST MODE FOR CARRYING OUT THE INVENTION

[0009]

The most important feature of the present invention is that Nb and Ta are selected as an additive element to an Fe-Co alloy represented by the composition formula regarding the atomic ratio: $\text{Fe}_X\text{-Co}_{100-X}$, wherein $10 \leq X \leq 80$, and as an optimal element to effectively achieve amorphization and improvement in corrosion resistance possibly by a single element when forming the soft magnetic film, and further, an optimal additive amount is found for achieving the described above effects.

[0010]

The Fe-Co alloy which is the base of the Fe-Co based alloy composition of the present invention is in a component range that represented by the composition formula regarding the atomic ratio: $\text{Fe}_X\text{-Co}_{100-X}$, wherein $10 \leq X \leq 80$. This is because an Fe-Co binary based alloy which is known to have the highest saturation magnetic moment among transition metal alloys shows the highest saturation magnetic moment when the atomic composition ratio of Fe to Co is around 65 to 35 (65:35) and a high saturation magnetic moment can be obtained in the case of an Fe-Co alloy having an atomic percent of Fe in the range of 10-80%.

If it is necessary to maximize the saturation magnetic moment, the atomic percent of Fe is preferably 50-80%, and if it is desired to lower magnetostriction as a thin film, the atomic percent of Fe is preferably 10-50%.

[0011]

The Fe-Co based alloy sputtering target material of the present invention comprises 10-20% by atom of one or two elements selected from Ta and Nb. This is because addition of Nb or Ta provides the effects that the Fe-Co based alloy is amorphized during sputtering, and at the same time the corrosion resistance is improved. Regarding the above
5 described effects, it is important to control it within 10-20% by atom since the corrosion resistance and the amorphization are not sufficient when it is less than 10% by atom and the magnetization is reduced when it is more than 20% by atom.

To maintain the amorphization, improvement in corrosion resistance and magnetization, it is more desirable that additive elements is one or two elements selected from Ta
10 and Nb in the range of 10-15% by atom.

In the Fe-Co based alloy sputtering target material of the present invention, the remainder other than Nb and Ta in the above described range is Fe and Co, and an unavoidable impurity. It is desirable that the impurity content is possibly little, and that gas components of oxygen and nitrogen are not more than 1000 ppm and unavoidable impurity elements such as Ni,
15 Si and Al other than the gas components are not more than 1000 ppm in total.
[0012]

According to the present invention, it is desirable that the Fe-Co based alloy sputtering target material have a maximum magnetic permeability of 250 or less.

In a magnetron sputtering method commonly used in formation of a soft magnetic
20 film, because the magnetron sputtering method is a method which makes rapid film formation possible by placing a magnet behind a target material to cause the magnetic flux to pass through the target material to its surface so that plasma converges in a pass-through-flux region, and is characterized by causing the magnetic flux to pass through the target material to the surface thereof, it becomes difficult to obtain the pass-through-flux necessary to cause the plasma on a
25 sputtering surface of the target material when the magnetic permeability of the target material itself is high. Accordingly, there are needs for reducing the magnetic permeability of a target material itself as much as possible.

Preferably, the maximum magnetic permeability of the Fe-Co based alloy sputtering target material of the present invention is possibly low. For stable sputtering
30 discharge in the magnetron sputtering method, it is preferably 250 or less, more preferably, 150 or less.
[0013]

According to the present invention, it is desirable that the PTF of the Fe-Co based alloy sputtering target material is 10% or more.

This PTF means a Pass-Through-Flux of a target material. The PTF is measured by a method of placing a permanent magnet behind a target material and measuring a flux passing through the target material onto a surface thereof, which method can quantitatively measure the pass-through-flux which is in a condition similar to a magnetron sputtering system.

5 An actual measurement is performed based on ASTM F1761-00 (Standard Test Method for Pass Through Flux of Circular Magnetic Sputtering Targets) and the PTF is determined by the following equation.

$$\text{(PTF)} = 100 \times (\text{flux strength in a condition where a target material is placed}) / (\text{flux strength in a condition where a target material is not placed}) (\%)$$

10 The PTF correlates with the thickness of a sputtering target, and the magnetron sputtering requires at least a certain value in order to stabilize the electric discharge during sputtering. By the investigation by the present inventors, it is confirmed that the stable electric discharge can be obtained in magnetron sputtering when the PTF is 10% or more.

[0014]

15 The Fe-Co based alloy sputtering target material of the present invention can be obtained by, for example, either adjusting alloy components so as to be in a desired composition range and then melting and casting it to obtain an Fe-Co based alloy raw material, or weighing and mixing Fe powder, Co powder, Ta powder or Nb powder into desired composition and then sintering the mixture to obtain a sintered body of an Fe-Co alloy, and subsequently by machining
20 it.

To obtain the above described low pass-through-flux and high PTF, it is desirable that, for example, when sintering Fe powder, Co powder, and Ta powder or Nb powder having been weighed and mixed in desired composition, the mixture is pressured and sintered at a pressure of 100 MPa or more and at a temperature of 1200°C or higher. This is because
25 interdiffusion of the mixed powder progresses by increasing the heating temperature during the pressurized sintering, so that diffusion of Ta and Nb into Fe and Co progresses, resulting in reduction of magnetization as an Fe-Co alloy target.

EXAMPLE 1

[0015]

30 The present invention will be further described in detail by the following examples.

First, Fe raw powder, Co raw powder, Ta raw powder and Nb raw powder each with a purity of 99.9% or more are prepared, and are weighed and mixed into mixed powders to

have the composition of each target material shown in Table 1 to make. The obtained mixed powders are each filled into mild steel capsules, and degassed and sealed, subsequently sintered by hot isostatic pressing to obtain a sintered body of 180 mm in diameter $\times$ 15 mm in thickness. The sintering is performed under conditions of a temperature of 1000°C, a pressure of 100 MPa and a holding time of 2 hours for Samples No. 1-7, and a temperature of 1250°C, a pressure of 100 MPa and a holding time of 2 hours for Samples No. 8-11. The obtained sintered bodies are each sliced into two target raw materials of 180 mm in diameter $\times$ 7 mm in thickness. With regard to Samples No. 1 and 2, one of the obtained target raw materials is processed to be 164 mm in diameter $\times$ 1 mm in thickness and brazed with In to a copper cooling plate of 180 mm in diameter $\times$ 4 mm in thickness to make an Fe-Co based alloy target material. With regard to Samples No. 3-11, one of the obtained target raw materials is processed to be 180 mm in diameter $\times$ 5 mm in thickness to make an Fe-Co based alloy sputtering target material.

[0016]

By the magnetron sputtering method using each target material obtained above, a thin film of 100 nm in thickness is formed on a glass substrate. The sputtering condition is of an Ar pressure of 0.6 Pa, and the making electric power is of 500 W.

The results of a corrosion resistance test in which each sample with a film is immersed in pure water for 24 hours are shown in Table 1. In Table 1, the samples with no corroded area are indicated by O and the samples with a corroded area are indicated by X.

[0017]

[Table 1]

Sample No.	Target composition (atom%)	Corrosion resistance evaluation	Remarks
1	(Fe ₆₅ -Co ₃₅)	×	Comparative example
2	(Fe ₆₅ -Co ₃₅) ₉₅ -Ta ₅	×	Comparative example
3	(Fe ₆₅ -Co ₃₅) _{88.5} -Ta _{11.5}	○	Example of the invention
4	(Fe ₆₅ -Co ₃₅) ₈₀ -Nb ₂₀	○	Example of the invention
5	(Fe ₆₅ -Co ₃₅) _{88.5} -Nb _{11.5}	○	Example of the invention
6	(Fe ₆₅ -Co ₃₅) _{85.5} -Nb _{14.5}	○	Example of the invention
7	(Fe ₆₅ -Co ₃₅) ₈₈ -Nb ₆ -Ta ₆	○	Example of the invention
8	(Fe ₃₀ -Co ₇₀) _{88.5} -Ta _{11.5}	○	Example of the invention
9	(Fe ₈₀ -Co ₂₀) _{88.5} -Ta _{11.5}	○	Example of the invention
10	(Fe ₆₅ -Co ₃₅) _{85.5} -Ta _{14.5}	○	Example of the invention
11	(Fe ₆₅ -Co ₃₅) ₈₀ -Ta ₂₀	○	Example of the invention

[0018]

As results of X-ray diffraction of Samples No. 3-11 which have shown good corrosion resistance, it is confirmed that a peak to be obtained from a crystalline phase is not observed, and the samples become amorphous. Further, the saturation magnetization of

5 Samples No. 3-11 is measured using a vibrating sample magnetometer VSM-3 made by TOEI INDUSTRY CO., LTD. The samples are measured by forming a thin film of 1 μm in thickness on an Si substrate by the magnetron sputtering method, then cutting out the substrate to a size of 10 mm $\times$ 10 mm and applying 795,775 (A/m) of an external magnetic field to it. The measured results are shown in Table 2.

10 [0019]

[Table 2]

Sample No.	Target composition (atom%)	Saturation magnetization (T)	Remarks
3	(Fe ₆₅ -Co ₃₅) _{88.5} -Ta _{11.5}	1.46	Example of the invention
4	(Fe ₆₅ -Co ₃₅) ₈₀ -Nb ₂₀	0.89	Example of the invention
5	(Fe ₆₅ -Co ₃₅) _{88.5} -Nb _{11.5}	1.78	Example of the invention
6	(Fe ₆₅ -Co ₃₅) _{85.5} -Nb _{14.5}	1.23	Example of the invention
7	(Fe ₆₅ -Co ₃₅) ₈₈ -Nb ₆ -Ta ₆	1.61	Example of the invention
8	(Fe ₃₀ -Co ₇₀) _{88.5} -Ta _{11.5}	1.41	Example of the invention
9	(Fe ₈₀ -Co ₂₀) _{88.5} -Ta _{11.5}	1.37	Example of the invention
10	(Fe ₆₅ -Co ₃₅) _{85.5} -Ta _{14.5}	1.15	Example of the invention
11	(Fe ₆₅ -Co ₃₅) ₈₀ -Ta ₂₀	0.50	Example of the invention

[0020]

From Tables 1 and 2, it can be seen that soft magnetic films formed by sputtering target materials comprising one or two elements selected from Ta and Nb of 10-20 atom% in an Fe-Co alloy have high corrosion resistance and saturation magnetization suitable for a soft

15 magnetic film of a magnetic recording medium. From Table 2, it can be seen that saturation magnetization of 1.1 or more can be obtained when a content of one or two elements selected from Ta and Nb to be added to an Fe-Co alloy is 10-15 atom%, and that it is desirable to control the amount of Ta and Nb in a target material within 10-15 atom% in order to obtain a soft magnetic film with higher saturation magnetization.

20 EXAMPLE 2

[0021]

Sintered bodies of 180 mm in diameter $\times$ 15 mm in thickness having the atomic

ratio composition of $(\text{Fe}_{65}\text{-Co}_{35})_{88.5}\text{-Ta}_{11.5}$ are made in a similar manner to EXAMPLE 1 except for the temperature condition of the hot isostatic pressing shown in Table 3. The sintered bodies are each sliced into two target raw materials of 180 mm in diameter $\times$ 7 mm in thickness. Sputtering target materials of 180 mm in diameter $\times$ 5 mm in thickness and of 180 mm in diameter $\times$ 6 mm in thickness are made from one of the raw materials obtained.

Specimens of 30 mm in length $\times$ 10 mm in width $\times$ 5 mm in thickness are collected from one of the target raw materials made above, and the magnetization curves of the specimens are measured using a DC magnetic properties measuring device TRF5A made by TOEI INDUSTRY CO., LTD. The Maximum magnetic permeability is determined from the obtained magnetization curves, and are shown in Table 3.

Further, the pass through flux (PTF) of each target material made above is measured, and is shown in Table 3. Actual measurements are performed based on ASTM F1761-00 (Standard Test Method for Pass Through Flux of Circular Magnetic Sputtering Targets) to determine the PTF by the following equation.

$$\text{PTF} = 100 \times (\text{flux strength in a condition where a target material is placed}) / (\text{flux strength in a condition where a target material is not placed}) (\%)$$

[0022]

[Table 3]

Sample No.	Temperature condition of hot isostatic pressing	Target thickness (mm)	Maximum magnetic permeability	PTF (%)
12	1000°C	5	210	10
13	1200°C	5	120	12
14	1250°C	5	75	15
15	1250°C	6	75	13

[0023]

From Table 3, it can be seen that the maximum magnetic permeability can be reduced and the PTF is improved by setting a higher temperature condition of hot isostatic pressing during pressurized sintering when making an Fe-Co based alloy sputtering target material of the present invention.

EXAMPLE 3

[0024]

Next, specimens of 30 mm in length, 10 mm in width and 5 mm in thickness are collected from one of the target raw materials of Samples No. 8-10 made in EXAMPLE 1. The

magnetization curves of these specimens are measured in a similar manner to EXAMPLE 2.

The maximum magnetic permeability is determined from the obtained magnetization curves, and are shown in Table 4.

The pass through flux (PTF) of each target material of Samples No. 8-10 is

5 measured, and is shown in Table 4. Actual measurements are performed in a similar manner to EXAMPLE 2.

[0025]

[Table 4]

Sample No.	Target composition (atom%)	Target thickness (mm)	Maximum magnetic permeability	PTF (%)
8	(Fe ₃₀ -Co ₇₀) _{88.5} -Ta _{11.5}	5	38	26
9	(Fe ₈₀ -Co ₂₀) _{88.5} -Ta _{11.5}	5	130	10
10	(Fe ₆₅ -Co ₃₅) _{85.5} -Ta _{14.5}	5	66	19

[0026]

From Table 4, it can be confirmed that the Fe-Co based alloy sputtering target

10 material of the present invention has sufficient PTF for electric discharge.

INDUSTRIAL APPLICABILITY

[0027]

The Fe-Co based alloy sputtering target material of the present invention is excellent in corrosion resistance and therefore applicable to producing of a stable perpendicular

15 magnetic recording medium.

CLAIMS

1. An Fe-Co based alloy sputtering target material for forming a soft magnetic film, comprising:
an Fe-Co alloy represented by the composition formula regarding an atomic ratio:
 $\text{Fe}_X\text{-Co}_{100-X}$, wherein $10 \leq X \leq 80$;
10-20 atom% of one or two elements selected from Ta and Nb; and
unavoidable impurity elements,
wherein the maximum magnetic permeability thereof is 150 or less and the PTF thereof is 10% or more.
2. The Fe-Co based alloy sputtering target material for forming a soft magnetic film according to claim 1, wherein the Fe-Co based alloy sputtering target material comprises 10-15 atom% of said one or two elements selected from Ta and Nb.