

SPUTTERING TARGET AND METHOD FOR MANUFACTURING SAME

BACKGROUND

Technical Field

[0001] The present invention relates to a sputtering target for forming, for example, a soft magnetic film of a magnetic recording medium, and to a method for manufacturing the sputtering target.

Related Art

[0002] In order to increase recording density of magnetic recording media, perpendicular magnetic recording has been put into practical use to replace conventional longitudinal magnetic recording. In recent years, since even higher recording density has been required, to realize this, it is necessary to reduce a crystal grain size of a recording layer so as to increase recording capacity per unit area.

However, when magnetic particles of a CoCrPt-SiO₂ recording layer used in the current perpendicular magnetic recording media are refined, there is a so-called thermal fluctuation problem that magnetically recorded data disappears under the influence of ambient heat. To suppress this problem, a material having high coercivity may be used in the recording layer. However, a problem arises in which a limit of a write magnetic field of a head may be exceeded so that recording becomes difficult.

As a method for solving these problems, for example, heat-assisted magnetic recording media have been proposed. During writing, the heat-assisted magnetic recording media use a method in which recording is performed while a recording layer is heated.

[0003] An FePt-based ordered alloy having high coercivity is promising as the recording layer of the heat-assisted magnetic recording media. However, to render an FePt-based alloy into ordered alloy, it is necessary to perform film formation accompanied by heating at 500°C to 700°C. During the film formation accompanied by heating, a soft magnetic film formed under the recording layer may also be heated at the same time, coarse crystal grains may be formed in the soft magnetic film, the coercivity may be increased, and there is a problem that recording characteristics may deteriorate.

[0004] In order to suppress this problem, for example, Patent Document 1 discloses a soft magnetic film, wherein the soft magnetic film has Fe as a main component and has specific amounts of Nb, Si, B and Cu added thereto in combination, thereby making the soft magnetic film amorphous or microcrystalline, suppressing coarsening of the crystal grains and reducing the coercivity.

In Patent Document 1, it is proposed that the soft magnetic film is formed by sputtering using a sputtering target (hereinafter simply referred to as “target”). The target used is manufactured by using, as raw material powder, powder in which pure metal powder and alloy powder are mixed so as to obtain a final composition of the soft magnetic film, and sintering the powder, or by a melting and casting method.

[Prior-Art Documents]

[Patent Documents]

[0005] Patent Document 1: JP 2013-84322

SUMMARY OF THE INVENTION

[Problems to Be Solved by the Invention]

[0006] The soft magnetic film disclosed in Patent Document 1 has Fe as the main component and has specific amounts of Nb, Si, B and Cu added thereto in combination, and therefore, an increase in coercivity can be suppressed even after the soft magnetic film is heated to a film formation temperature of the recording layer. In this respect, the soft magnetic film is a useful technique.

According to a study by the present inventors, in a target made by the manufacturing method disclosed in Patent Document 1, which has Fe as the main component and has specific amounts of Nb, Si, B and Cu added thereto in combination, a mechanical strength, particularly bending strength, is confirmed to be low. There are fears that the problem of reduction in bending strength may cause cracks in the case where an impact is received during machining such as cutting or blast-machining of the target or during chucking of the target to a sputter device.

An object of the present invention is to provide a target capable of suppressing cracks in the target and suitable for forming, for example, a soft magnetic film of a heat-assisted magnetic recording medium, and a method for manufacturing the target.

[Means for Solving the Problems]

[0007] The present invention includes a sputtering target, the sputtering target having a composition formula expressed as $\text{Fe}_{100-a-b-c}\text{-Cu}_a\text{-Si}_b\text{-M}_c$ in terms of atomic ratio, wherein $0.1 \leq a \leq 5.0$, $10.0 \leq b \leq 20.0$, $10.0 \leq c \leq 25.0$, and M is one or more elements selected from Nb and B, containing a remainder composed of unavoidable impurities, and having a bending strength of 500 MPa or more.

In addition, in the sputtering target of the present invention, a number of Fe phases having an inscribed circle diameter of 1 μm or greater is preferably less than 1.0 per 60000 μm^2 .

[0008] The sputtering target of the present invention can be obtained by pressure-sintering raw material powder, wherein the raw material powder has a composition formula expressed as $\text{Fe}_{100-a-b-c}\text{-Cu}_a\text{-Si}_b\text{-M}_c$ in terms of atomic ratio, wherein $0.1 \leq a \leq 5.0$, $10.0 \leq b \leq 20.0$, $10.0 \leq c \leq 25.0$, and M is one or more elements selected from Nb and B, and contains a remainder composed of unavoidable impurities.

[Effects of the Invention]

[0009] The present invention is capable of suppressing the occurrence of cracks in the target during machining or chucking, and is therefore a useful technique for manufacturing a soft magnetic film of a magnetic recording medium.

BRIEF DESCRIPTION OF THE DRAWINGS

[0010] FIG. 1 is a schematic diagram of a metal structure of a target of Example 1 of the present invention.

FIG. 2 is a schematic diagram of a metal structure of a target of Example 2 of the present invention.

FIG. 3 is a schematic diagram of a metal structure of a target of Example 3 of the present invention.

FIG. 4 is a schematic diagram of a metal structure of a target of Comparative Example 1.

FIG. 5 is a schematic diagram of a metal structure of a target of Comparative Example 2.

FIG. 6 is a schematic diagram of a metal structure of a target of Comparative Example 3.

FIG. 7 is a schematic diagram of a metal structure of a broken portion of the target

of Comparative Example 1.

DESCRIPTION OF THE EMBODIMENTS

[0011] A target of the present invention contains Cu in the range of 0.1 atomic percent (at.%) to 5.0 at.%. Since Cu has a positive mixing enthalpy with Fe, when a soft magnetic film is heated, Cu forms a cluster, which becomes a starting point of formation of a microcrystalline nucleus. Accordingly, microcrystalline grains are uniformly dispersed in the soft magnetic film, and coercivity of the soft magnetic film after heating can be reduced. To form the starting point of such a microcrystalline nucleus, it is necessary that Cu be contained at 0.1 at.% or more. Hence, in the target of the present invention, the lower limit of the content of Cu is set to 0.1 at.%. In addition, for the same reason as above, the content of Cu is preferably set to 0.8 at.% or more.

On the other hand, when the content of Cu exceeds 5.0 at.%, excessive Cu may be precipitated onto a surface of the soft magnetic film, thus increasing surface roughness. The increase in surface roughness of the soft magnetic film causes an increase in noises of a recording medium. Hence, in the target of the present invention, the upper limit of the content of Cu is set to 5.0 at.%. In addition, for the same reason as above, the content of Cu is preferably set to 1.2 at.% or less.

[0012] To reduce the coercivity of the soft magnetic film, it is necessary to reduce magnetostriction. In the soft magnetic film formed using the target of the present invention, a structure is obtained in which the microcrystalline grains are dispersed in a remaining amorphous phase due to heating.

The microcrystalline grains are composed of FeSi solid solutions and have

negative magnetostriction. Meanwhile, the remaining amorphous phase is composed of FeNbSiB and has positive magnetostriction. Hence, in the soft magnetic film formed by the target of the present invention, the magnetostrictions of the microcrystalline grains and the remaining amorphous phase are canceled out by each other, the magnetostriction can be reduced, and a reduction in the coercivity can be realized.

The magnitude of magnetostriction of the microcrystalline grains may be adjusted by the content of Si. In the target of the present invention, the content of Si is set to 10.0 at.% or more. Accordingly, the microcrystalline grains having negative magnetostriction are increased, and the magnetostriction can be reduced. In addition, for the same reason as above, the content of Si is preferably set to 13.0 at.% or more.

On the other hand, in the target of the present invention, the content of Si is set to 20.0 at.% or less. Accordingly, excessive generation of the FeSi solid solutions is suppressed, and the magnetostriction can be reduced. In addition, for the same reason as above, the content of Si is preferably set to 15.8 at.% or less.

[0013] As mentioned above, in the soft magnetic film formed by the target of the present invention, a structure is obtained in which the microcrystalline grains are dispersed in the remaining amorphous phase. Nb and B have effects of stabilizing the remaining amorphous phase and suppressing the coarsening of microcrystalline grains caused by heating.

In the target of the present invention, a total content of an M element, namely one or more elements selected from Nb and B, is set to 10.0 at.% or more. Accordingly, the coarsening of the microcrystalline grains can be suppressed, and a soft magnetic film having low coercivity can be obtained. In addition, for the same reason as above,

the total content of the M element is preferably set to 11.0 at.% or more. The content of Nb is preferably set to 2.5 at.% or more, and the content of B is preferably set to 8.5 at.% or more.

On the other hand, in the target of the present invention, the total content of the M element is set to 25.0 at.% or less. Accordingly, a soft magnetic film having high saturation magnetic flux density can be obtained. In addition, from the viewpoint of suppressing a brittle intermetallic compound that affects the bending strength, the total content of the M element is preferably set to 16.0 at.% or less. The content of Nb is preferably set to 6.5 at.% or less, and the content of B is preferably set to 9.5 at.% or less.

[0014] In the target of the present invention, a remainder other than the above-described Cu, Si, Nb and B is composed of Fe and unavoidable impurities. This is to improve the saturation magnetic flux density which is one of the characteristics required for the soft magnetic film. Moreover, in order to improve the saturation magnetic flux density, a part of Fe may be replaced with one or more elements selected from Co and Ni.

[0015] The target of the present invention has a bending strength of 500 MPa or more. Accordingly, in a manufacturing process of the target, the occurrence of cracks during machining or chucking can be suppressed. The bending strength of the target of the present invention is preferably 800 MPa or more, more preferably 1000 MPa or more.

In the target of the present invention, a number of Fe phases having an inscribed circle diameter of 1 μm or greater is preferably set to less than 1.0 per 60000 μm^2 . This is because that, when 1.0 or more Fe phases having an inscribed circle diameter of

1 μm or greater exist in a metal structure of the target, grain boundaries of the Fe phases are likely to become a starting point of cracks in the target.

Moreover, the inscribed circle diameter of the Fe phase mentioned in the present invention can be measured by capturing an image of the Fe phase shown in light gray color in a backscattered electron image by a scanning electron microscope in an arbitrary visual field of $60000\ \mu\text{m}^2$ on a surface of the target which serves as a sputtering surface. The number of Fe phases having an inscribed circle diameter of 1 μm or greater can be obtained by observing one visual field of $60000\ \mu\text{m}^2$ on the surface serving as the sputtering surface and counting the number of the Fe phases having an inscribed circle diameter of 1 μm or greater existing in the visual field. In addition, from the viewpoint of suppressing cracks in the entire target, the number of the Fe phases having an inscribed circle diameter of 1 μm or greater may be obtained by observing a plurality of visual fields (for example, five visual fields) each amounting to $60000\ \mu\text{m}^2$ on the surface serving as the sputtering surface, counting the number of the Fe phases having an inscribed circle diameter of 1 μm or greater existing in each of the visual fields and calculating an average value thereof.

[0016] The target of the present invention can be obtained by pressure-sintering raw material powder, wherein the raw material powder has a composition formula expressed as $\text{Fe}_{100-a-b-c}\text{-Cu}_a\text{-Si}_b\text{-M}_c$ in terms of atomic ratio, wherein $0.1 \leq a \leq 5.0$, $10.0 \leq b \leq 20.0$, $10.0 \leq c \leq 25.0$, and M is one or more elements selected from Nb and B, and contains a remainder composed of unavoidable impurities.

As mentioned above, in the target disclosed in Patent Document 1, which is obtained by sintering the raw material powder in which pure metal powder and alloy powder are mixed so as to obtain the final composition of the soft magnetic film, a

coarse Fe phase emerges as in the schematic diagram of the metal structure of the broken portion of the target shown in FIG. 7. As a region of the Fe phase increases, a grain boundary thereof becomes the starting point of cracks in the target.

In contrast, in the present invention, the raw material powder having a composition formula expressed as $\text{Fe}_{100-a-b-c}\text{-Cu}_a\text{-Si}_b\text{-M}_c$ in terms of atomic ratio, wherein $0.1 \leq a \leq 5.0$, $10.0 \leq b \leq 20.0$, $10.0 \leq c \leq 25.0$, and M is one or more elements selected from Nb and B, and containing a remainder composed of unavoidable impurities, i.e., the raw material powder including the final composition of the target, is pressure-sintered. Accordingly, the emergence of the Fe phase can be suppressed, and the occurrence of cracks during machining or chucking of the target can be suppressed.

[0017] In the present invention, the raw material powder including the final composition of the target, which is for pressure-sintering, can be made by, for example, a method of pulverizing an ingot obtained by casting a molten alloy adjusted to have the final composition, or a gas atomization method using the molten alloy adjusted to have the final composition. Among them, it is preferable to use the gas atomization method by which spherical powder having a small amount of impurities mixed therein, having a high filling rate, and suitable for pressure-sintering can be obtained. Herein, to suppress oxidation of the raw material powder, it is preferable to use an Ar gas or a nitrogen gas being an inert gas, as an atomizing gas.

The pressure-sintering is preferably performed under the conditions of a temperature of 700°C to 1050°C, a pressure of 100 MPa to 200 MPa and a duration of 1 hour to 10 hours.

By setting the sintering temperature to 700°C or higher, sintering of raw material powder containing Nb being a high melting metal is allowed to proceed, and the

occurrence of voids can be suppressed. In addition, by setting the sintering temperature to 1050°C or lower, melting of the raw material powder can be suppressed.

In addition, by setting the pressure to 100 MPa or more, the sintering is accelerated and the occurrence of voids can be suppressed. In addition, by setting the pressure to 200 MPa or less, introduction of residual stress to the target during sintering is suppressed, and the occurrence of cracks in a sintered body can be suppressed.

In addition, by setting the sintering holding time to 1 hour or longer, the sintering is accelerated and the occurrence of voids can be suppressed. In addition, by setting the sintering holding time to 10 hours or shorter, the manufacturing can be performed by suppressing growth of a brittle Nb-containing intermetallic compound without reducing manufacturing efficiency.

[Examples]

[0018] Raw material powder of under 80 mesh was prepared using a raw material piece composed of a pure metal having purity of 99.9% or higher by vacuum-melting a molten alloy having an alloy composition having a composition formula expressed as $\text{Fe}_{73.5}\text{-Cu}_{1.0}\text{-Si}_{13.5}\text{-Nb}_{3.0}\text{-B}_{9.0}$ in terms of atomic ratio, producing powder by a gas atomization method using an Ar gas, and removing coarse powder by classification.

Then, the raw material powder was filled in a soft steel capsule and then degassed and sealed, followed by hot isostatic pressing under the conditions of a temperature of 950°C, a pressure of 122 MPa, and a holding time of 1 hour to obtain a sintered body. The sintered body was subjected to machining to produce a target serving as Example 1 of the present invention.

[0019] Raw material powder of under 80 mesh was prepared using a raw material piece composed of a pure metal having purity of 99.9% or higher by vacuum-melting a

molten alloy having an alloy composition having a composition formula expressed as $\text{Fe}_{65.5}\text{-Cu}_{1.0}\text{-Si}_{13.5}\text{-Nb}_{5.0}\text{-B}_{15.0}$ in terms of atomic ratio, producing powder by a gas atomization method using an Ar gas, and removing coarse powder by classification.

Then, the raw material powder was filled in a soft steel capsule and then degassed and sealed, followed by hot isostatic pressing under the conditions of a temperature of 950°C, a pressure of 122 MPa, and a holding time of 1 hour to obtain a sintered body. The sintered body was subjected to machining to produce a target serving as Example 2 of the present invention.

[0020] Raw material powder of under 80 mesh was prepared using a raw material piece composed of a pure metal having purity of 99.9% or higher by vacuum-melting a molten alloy having an alloy composition having a composition formula expressed as $\text{Fe}_{57.0}\text{-Cu}_{3.0}\text{-Si}_{20.0}\text{-Nb}_{5.0}\text{-B}_{15.0}$ in terms of atomic ratio, producing powder by a gas atomization method using an Ar gas, and removing coarse powder by classification.

Then, the raw material powder was filled in a soft steel capsule and then degassed and sealed, followed by hot isostatic pressing under the conditions of a temperature of 950°C, a pressure of 122 MPa, and a holding time of 1 hour to obtain a sintered body. The sintered body was subjected to machining to produce a target serving as Example 3 of the present invention.

[0021] Raw material powder was produced using pure metal powders of Fe, Nb and Cu having purity of 99.9% or higher as well as metalloid powders of Si and B by mixing the powders so as to obtain an alloy composition having a composition formula expressed as $\text{Fe}_{73.5}\text{-Cu}_{1.0}\text{-Si}_{13.5}\text{-Nb}_{3.0}\text{-B}_{9.0}$ in terms of atomic ratio.

Then, the raw material powder was filled in a soft steel capsule and then degassed and sealed, followed by hot isostatic pressing under the conditions of a temperature of

750°C, a pressure of 122 MPa, and a holding time of 1 hour to obtain a sintered body. The sintered body was subjected to machining to produce a target serving as Comparative Example 1.

[0022] Raw material powder was produced using pure metal powders of Fe, Nb and Cu having purity of 99.9% or higher as well as metalloid powders of Si and B by mixing the powders so as to obtain an alloy composition having a composition formula expressed as $\text{Fe}_{65.5}\text{-Cu}_{1.0}\text{-Si}_{13.5}\text{-Nb}_{5.0}\text{-B}_{15.0}$ in terms of atomic ratio.

Then, the raw material powder was filled in a soft steel capsule and then degassed and sealed, followed by hot isostatic pressing under the conditions of a temperature of 950°C, a pressure of 122 MPa, and a holding time of 1 hour to obtain a sintered body. The sintered body was subjected to machining to produce a target serving as Comparative Example 2.

[0023] Raw material powder was produced using pure metal powders of Fe, Nb and Cu having purity of 99.9% or higher as well as metalloid powders of Si and B by mixing the powders so as to obtain an alloy composition having a composition formula expressed as $\text{Fe}_{57.0}\text{-Cu}_{3.0}\text{-Si}_{20.0}\text{-Nb}_{5.0}\text{-B}_{15.0}$ in terms of atomic ratio.

Then, the raw material powder was filled in a soft steel capsule and then degassed and sealed, followed by hot isostatic pressing under the conditions of a temperature of 950°C, a pressure of 122 MPa, and a holding time of 1 hour to obtain a sintered body. The sintered body was subjected to machining to produce a target serving as Comparative Example 3.

[0024] In order to confirm bending strength of each of the above-obtained targets in a thickness direction, a bending test piece of 3 mm×4 mm×25 mm was sampled. A three-point bending test was carried out on the sampled test piece under the conditions

of a crosshead speed of 0.5 mm/min and a distance of 20 mm between fulcrums. A maximum bending load was measured from an obtained load-deflection curve, and the bending strength was calculated. Results thereof are shown in Table 1.

[0025] In addition, in order to observe a surface serving as a sputtering surface of each of the above-obtained targets, a test piece of 10 mm×10 mm×5 mm was sampled and a surface thereof serving as the sputtering surface was polished, followed by observing a metal structure of the target using a scanning electron microscope. The observation was performed in the following manner. In a backscattered electron image of the surface serving as the sputtering surface of each sample under the scanning electron microscope, among arbitrary visual fields, one visual field which amounts to 60000 μm^2 was observed, an inscribed circle diameter of Fe phases existing within the visual field was measured, and the number of the Fe phases having an inscribed circle diameter of 1 μm or greater was counted. Results thereof are shown in Table 1.

In addition, for each sample, in the sample having Fe phases existing in the metal structure, the diameter of an inscribed circle drawn within the region of each Fe phase was measured. The diameter of the maximum inscribed circle in each Fe phase was shown as the Maximum Inscribed Circle Diameter of Fe Phase in Table 1. In addition, the measurement examples are shown in FIG. 1 to FIG. 6.

[0026] [Table 1]

	Composition of Target [At.%]	Bending Strength [MPa]	Fe Phase	
			Number of Fe Phases Having Inscribed Circle Diameter of 1 μm or Greater	Maximum Inscribed Circle Diameter [μm]
Example 1	Fe _{73.5} -Cu _{1.0} -Si _{13.5} -Nb _{3.0} -B _{9.0}	1105	0	0 (No Fe phase)
Example 2	Fe _{65.5} -Cu _{1.0} -Si _{13.5} -Nb _{5.0} -B _{15.0}	784	0	0 (No Fe phase)
Example 3	Fe _{57.0} -Cu _{3.0} -Si _{20.0} -Nb _{5.0} -B _{15.0}	772	0	0 (No Fe phase)
Comparative Example 1	Fe _{73.5} -Cu _{1.0} -Si _{13.5} -Nb _{3.0} -B _{9.0}	487	2.0	31
Comparative Example 2	Fe _{65.5} -Cu _{1.0} -Si _{13.5} -Nb _{5.0} -B _{15.0}	345	4.0	33
Comparative Example 3	Fe _{57.0} -Cu _{3.0} -Si _{20.0} -Nb _{5.0} -B _{15.0}	280	2.0	22

[0027] In all the targets of Comparative Example 1 to Comparative Example 3, the bending strength was a low value less than 500 MPa. In the targets of Comparative Example 1 to Comparative Example 3, when observing an arbitrary unit area of 60000 μm^2 , it was confirmed that the number of the Fe phases having an inscribed circle diameter of 1 μm or greater was 2.0 or more, as shown in FIG. 4 to FIG. 6. In addition, in all the targets of Comparative Example 1 to Comparative Example 3, the diameter of the maximum inscribed circle drawn within the region of the Fe phase exceeded 20 μm .

On the other hand, it can be confirmed that all the targets of the present invention have a high bending strength of 500 MPa or more. In the targets of the present

invention, when observing an arbitrary unit area of $60000\ \mu\text{m}^2$, it can be confirmed that the number of the Fe phases having an inscribed circle diameter of $1\ \mu\text{m}$ or greater was less than 1.0, as shown in FIG. 1 to FIG. 3. Accordingly, in the targets of the present invention, the occurrence of cracks during machining or chucking can be suppressed.

WHAT IS CLAIMED IS:

1. A sputtering target, having a composition formula expressed as $\text{Fe}_{100-a-b-c}\text{-Cu}_a\text{-Si}_b\text{-M}_c$ in terms of atomic ratio, wherein $0.1 \leq a \leq 5.0$, $10.0 \leq b \leq 20.0$, $10.0 \leq c \leq 25.0$, and M is one or more elements selected from Nb and B, containing a remainder composed of unavoidable impurities, and having a bending strength of 500 MPa or more.

2. The sputtering target according to claim 1, wherein a number of Fe phases having an inscribed circle diameter of 1 μm or greater is less than 1.0 per 60000 μm^2 .

3. A method for manufacturing a sputtering target, comprising pressure-sintering raw material powder, wherein the raw material powder has a composition formula expressed as $\text{Fe}_{100-a-b-c}\text{-Cu}_a\text{-Si}_b\text{-M}_c$ in terms of atomic ratio, wherein $0.1 \leq a \leq 5.0$, $10.0 \leq b \leq 20.0$, $10.0 \leq c \leq 25.0$, and M is one or more elements selected from Nb and B, and contains a remainder composed of unavoidable impurities.