

FERROMAGNETIC MATERIAL SPUTTERING TARGET

TECHNICAL FIELD

[0001]

The present invention relates to a Co-Pt-based ferromagnetic material sputtering target suitable for forming a magnetic thin film in a magnetic recording medium.

BACKGROUND ART

[0002]

In the field of magnetic recording typified by hard disk drives, a material based on Co, which is a ferromagnetic metal, is used as the material of a magnetic thin film employed for recording.

[0003]

From the viewpoint of high productivity, magnetic thin films are often produced by sputtering a sputtering target containing the above-mentioned material as a component with a DC magnetron sputtering apparatus. However, when attempting to sputter a ferromagnetic material sputtering target with a magnetron sputtering apparatus, most of the magnetic flux from the magnet passes through the interior of the ferromagnetic target, which reduces flux leakage, causing the problem that discharge does not occur during sputtering or that even if there is discharge, the discharge is not stable. To solve this problem, the content ratio of Co, which is a ferromagnetic metal, may be reduced. However, reducing the amount of Co does not solve the essential problem because it does not enable the desired magnetic recording film to be obtained. Although flux leakage can be improved by reducing

the thickness of the target, in this case the target life is shortened, and it becomes necessary to frequently replace the target, which causes costs to increase.

[0004]

Therefore, conventionally, a technique has been employed in which the Cr ratio is increased to partially make it non-magnetic, thereby increasing flux leakage. For example, for a recording layer of a hard disk employing an in-plane magnetic recording system, Co-Cr-based ferromagnetic alloys or Co-Cr-Pt-based ferromagnetic alloys containing Co as a main component have been used. Further, for the recording layer of a hard disk employing a perpendicular magnetic recording system, which have been developed for practical use in recent years, a composite material is often used in which non-magnetic particles, such as an oxide or carbon, are dispersed in a Co-Cr-Pt-based ferromagnetic alloy having Co as a main component. However, the current mainstream composition is a composition with little or no Cr, and it is difficult to sufficiently obtain the effect of an increase in flux leakage with the conventional methods.

[0005]

In view of such a background, in WO 2012/077665, there is proposed a ferromagnetic material sputtering target characterized by having, in a sputtering target composed of metals having a composition of 5 mol% or more of Pt, 20 mol% or less of Cr, and a balance of Co, a metal base (A) and, in the metal base (A), a Co-Pt alloy phase (B) containing 40 to 76 mol% of Pt. According to WO 2012/077665, if the diameter of the Co-Pt alloy phase (B) is too small, diffusion between the metal base (A) and the phase (B) progresses and the difference between the constituent elements becomes unclear, and hence it is preferable for the diameter to be 10 μm or more. Further, according to WO 2012/077665, if the diameter of the Co-Pt alloy phase (B) is too large, the problem of particle matter

during sputtering tends to occur, and hence it is desirable for the diameter to be 150 μm or less.

[0006]

In addition, in WO 2012/081669, there is proposed a ferromagnetic material sputtering target characterized by having, in a sputtering target composed of metals having a composition of 20 mol% or less of Cr, 5 mol% or more of Pt, and a balance of Co, a metal base (A) and, in the metal base (A), a Co-Pt alloy phase (B) containing 40 to 76 mol% of Pt and a metal or alloy phase (C) containing Co or Co as a main component different from the phase (B). According to WO 2012/081669, if the diameter of the Co-Pt alloy phase (B) is too small, diffusion between the metal base (A) and the phase (B) progresses and the difference between the constituent elements becomes unclear, and hence it is preferable for the diameter to be 10 μm or more. Further, according to WO 2012/081669, if the diameter of the Co-Pt alloy phase (B) is too large, the problem of particle matter during sputtering tends to occur, and hence it is desirable for the diameter to be 150 μm or less.

CITATION LIST

[Patent Literature]

[0007]

[Patent Literature 1] WO2012/077665

[Patent Literature 2] WO2012/081669

SUMMARY OF INVENTION

Technical Problem

[0008]

As described in Patent Literature 1 and Patent Literature 2, a ferromagnetic material sputtering target having a Co-Pt alloy phase in a metal base provides the

advantage that the flux leakage can be increased. However, according to the results of studies carried out by the present inventor, there is still room for improvement regarding the suppression of particle matter during sputtering of the sputtering target. Accordingly, one object of the present invention is to provide a Co-Pt ferromagnetic material sputtering target that has a high flux leakage and is capable of suppressing generation of particle matter during sputtering.

Solution to Problem

[0009]

In Patent Literature 1 and Patent Literature 2, it is taught that the Co-Pt alloy phase should be a phase having a diameter of 10 μm or more, but it was learned that such a coarse Co-Pt alloy phase causes particle matter to be generated during sputtering. In view of this, the present inventor extensively investigated effective means for suppressing the particle matter while utilizing the advantages of a Co-Pt alloy phase capable of increasing flux leakage. As a result, the present inventor discovered that a method in which the Co phase is coarsened while making the Co-Pt alloy phase finer is effective, and based on this knowledge, completed the present invention.

[0010]

In one aspect, the present invention is A ferromagnetic material sputtering target comprising a total of 70 mol% or more of metal Co and metal Pt in a molar ratio of $\text{Co:Pt} = \text{X:}100 - \text{X}$ ($59 \leq \text{X} < 100$) and comprising 0 mol% or more and 20 mol% or less of metal Cr,

wherein the ferromagnetic material sputtering target comprises:

a Co particle phase comprising 90 mol% or more of metal Co and having an average particle size of 30 to 300 μm ; and

a Co-Pt alloy particle phase that, under a condition of a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$), comprises a total of 70 mol% or more of metal Co and metal Pt and has an average particle size of 7 μm or less.

[0011]

In one embodiment of the ferromagnetic material sputtering target according to the present invention, the ferromagnetic material sputtering target comprises a total of 30 mol% or less of one or two or more third elements selected from a group consisting of B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Al, Si, and Zn.

[0012]

In another embodiment of the ferromagnetic material sputtering target according to the present invention, the ferromagnetic material sputtering target comprises a total of 25 mol% or less of one or two or more non-magnetic materials selected from a group consisting of carbon, an oxide, a nitride, a carbide, and a carbonitride.

[0013]

In yet another embodiment of the ferromagnetic material sputtering target according to the present invention, the ferromagnetic material sputtering target has peaks at positions $2\theta = 33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$, respectively, in XRD measurement with a 2θ measurement range of 30° to 60° .

[0014]

In another aspect, the present invention is a method of producing a ferromagnetic material sputtering target, wherein the method comprises:

a step of providing a Co powder comprising 90 mol% or more of metal Co and having a median size of 30 to 300 μm ;

a step of providing a Co-Pt alloy powder that, under a condition of a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$), comprises a total of 70 mol% or more of metal Co and metal Pt and has a median size of 7 μm or less;

a step of obtaining a mixed powder by mixing the Co-Pt alloy powder and the Co powder such that, under a condition of a molar ratio of Co:Pt = X:100 - X ($59 \leq X < 100$), the mixed powder comprises a total of 70 mol% or more of metal Co and metal Pt and comprises 0 mol% or more and 20 mol% or less of metal Cr; and
a step of firing the mixed powder.

[0015]

In one embodiment of the method of producing a ferromagnetic material sputtering target according to the present invention, the step of obtaining the mixed powder further comprises mixing one or two or more powders selected from a group consisting of B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Al, Si, and Zn.

[0016]

In another embodiment of the method of producing a ferromagnetic material sputtering target according to the present invention, the step of obtaining the mixed powder further comprises mixing one or two or more non-magnetic materials selected from a group consisting of carbon, an oxide, a nitride, a carbide, and a carbonitride.

[0017]

In yet another embodiment of the method of producing a ferromagnetic material sputtering target according to the present invention, the step of providing the Co-Pt alloy powder that has the median size of 7 μm or less comprises:

a step of obtaining a first sintered compact by firing a mixed powder of a Co powder and a Pt powder in a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$) at a heating temperature of 800°C to 1000°C such that the first sintered compact has peaks at positions $2\theta = 33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$, respectively, in XRD measurement with a 2θ measurement range of 30° to 60°; and

a step of grinding the first sintered compact.

[0018]

In still yet another aspect, the present invention is a method of producing a magnetic recording film comprising using the sputtering target according to the present invention.

Advantageous Effects of Invention

[0019]

The ferromagnetic material sputtering target of the present invention serves as a target with a large amount of flux leakage, and when used in a magnetron sputtering apparatus, the ionization promotion of the inert gas proceeds efficiently and stable discharge is obtained. According to the present invention, since the thickness of the target can be increased, there is an advantageous effect in that the replacement frequency of the target is reduced, which enables the magnetic thin film to be manufactured at low cost. In addition, with the ferromagnetic material sputtering target of the present invention, there is less particle matter at the time of sputtering, and stable sputtering becomes possible.

Brief Description of Drawings

[0020]

[Figure 1] Figure 1 illustrates an XRD profile of Comparative Example 1.

[Figure 2] Figure 2 illustrates an XRD profile of Comparative Example 2.

[Figure 3] Figure 3 illustrates an XRD profile of Comparative Example 3.

[Figure 4] Figure 4 illustrates an XRD profile of Example 1.

[Figure 5] Figure 5 illustrates an XRD profile of Example 2

[Figure 6] Figure 6 illustrates an XRD profile of Example 3.

[Figure 7] Figure 7 illustrates an XRD profile of Example 5.

[Figure 8] Figure 8 illustrates an XRD profile of Example 7.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0021]

(1. Overall Composition)

In one embodiment, the ferromagnetic material sputtering target according to the present invention comprises, under a condition of a molar ratio of Co:Pt = X:100 - X ($59 \leq X < 100$), a total of 70 mol% or more of metal Co and metal Pt and 0 mol% or more and 20 mol% or less of metal Cr. In this composition, it is generally difficult to obtain a high flux leakage because the content of the metal Co, which is a ferromagnetic material, is large and the content of the metal Cr, which has an effect of increasing the flux leakage, is small. According to the present invention, it is possible for a sputtering target having a composition that does not easily obtain a high flux leakage to increase the flux leakage and further reduce particle matter by, designing the structure of the target in a special manner.

[0022]

For the ferromagnetic material sputtering target according to the present invention, in consideration of hard disk media production applications, in one embodiment X is $70 \leq X \leq 90$, and in another embodiment X is $80 \leq X \leq 85$.

[0023]

In one embodiment, the ferromagnetic material sputtering target according to the present invention may contain a total of 75 mol% or more of metal Co and metal Pt, and in another embodiment it may contain a total of 85 mol% or more of metal Co and metal Pt. Further, in one embodiment, the ferromagnetic material sputtering target according to the present invention may contain a total of 95 mol% or less of metal Co and metal Pt, and in another embodiment it may contain a total of 90 mol% or less of metal Co and metal Pt. A ferromagnetic material sputtering

target containing a total of 75 to 95 mol% of metal Co and metal Pt is suitable for perpendicular magnetic recording films.

[0024]

In one embodiment, the ferromagnetic material sputtering target according to the present invention may contain 0 mol% or more and 15 mol% or less of metal Cr, in another embodiment it may contain 0 mol% or more and 10% mol or less of metal Cr, in yet another embodiment it may contain 0 mol% or more and 5 mol% or less of metal Cr, in still yet another embodiment it may contain 0 mol% or more and 1 mol% or less of metal Cr, and in still yet another embodiment it does not contain metal Cr.

[0025]

(2. Co-Pt Alloy Particle Phase)

In order to increase the flux leakage in a sputtering target having such a Co-rich composition, it is desirable for the sputtering target to have a Co-Pt alloy particle phase that contains, in terms of a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$), a total of 70 mol% or more of metal Co and metal Pt, preferably 80 mol% or more, more preferably 90 mol% or more, and still more preferably 99 mol% or more. The Co-Pt alloy particle phase forms an ordered phase having a $L1_0$ structure, and can exhibit high magnetic anisotropy, and hence contributes to an increase in flux leakage. From the viewpoint of increasing the flux leakage, Y is preferably $25 \leq Y \leq 55$, more preferably $34.5 \leq Y \leq 55$, and still more preferably $40 \leq Y \leq 50$. The Co-Pt alloy particle phase can be present dispersed in the target structure.

[0026]

Although the Co-Pt alloy particle phase contributes to an improvement of the flux leakage, when the average particle size is large, the large particle size becomes a factor causing an increase in particle matter. In Patent Literature 1 and Patent Literature 2, the problem of particle matter during sputtering caused by the Co-Pt alloy particle phase is said to occur when the diameter exceeds 150 μm , and it is

desirable for the Co-Pt alloy particle phase to be as fine as possible. Therefore, in the ferromagnetic material sputtering target according to the present invention, the average particle size of the Co-Pt alloy particle phase is preferably 7 μm or less, more preferably 5 μm or less, and still more preferably 2 μm or less. However, if the average particle size of the Co-Pt alloy particle phase is too small, the Co-Pt alloy particle phase tends to diffuse into other phases during sintering, which increases the likelihood of the $L1_0$ structure being lost. Therefore, the average particle size of the Co-Pt alloy particle phase is preferably 0.1 μm or more, more preferably 0.2 μm or more, and still more preferably 0.5 μm or more.

[0027]

From the viewpoint of enhancing the effect of increasing the flux leakage, the Co-Pt alloy particle phase is, preferably 20% by mass or more, more preferably 30% by mass or more, and still more preferably 40 % by mass or more, based on the total mass of the sputtering target. From the viewpoint of finely dispersing the non-magnetic material, the Co-Pt alloy particle phase is preferably 70% by mass or less, more preferably 65% by mass or less, and still more preferably 60% by mass or less, based on the total mass of the sputtering target.

[0028]

(3. Co Particle Phase)

In one embodiment, the sputtering target with a Co-rich composition according to the present invention has, in addition to the Co-Pt alloy particle phase, a Co particle phase containing 90 mol% or more of metal Co. In order to increase the ratio of the Co-Pt alloy particle phase included in the target, the Co particle phase preferably contains 95 mol% or more of metal Co, and more preferably 99.9 mol% or more. The Co particle phase can be present dispersed in the target structure.

[0029]

When the Co-Pt alloy particle phase is made finer, if the Co particle phase is also made finer, the contact points with the Co-Pt alloy particle phase increase, which promotes the diffusion of the constituent elements during sintering, causing the problem that the $L1_0$ structure is lost. Therefore, it is desirable that the Co particle phase is conversely made relatively coarse. Specifically, the average particle size of the Co particle phase containing 90 mol% or more of metal Co is preferably 30 μm or more, more preferably 50 μm or more, and still more preferably 100 μm or more. However, because the average particle size of the Co particle phase may cause particle matter to be generated if it is too large, it is preferable for the average particle size to be 300 μm or less, more preferably 200 μm or less, and still more preferably 150 μm or less.

[0030]

From the viewpoint of further suppressing the diffusion of the Co-Pt alloy particle phase during sintering in order to increase the flux leakage, the Co particle phase is preferably 15% by mass or more, more preferably 25% by mass or more, and still more preferably 30% by mass or more, based on the total mass of the sputtering target. In addition, because the Co particle phase particles are coarse, the Co particle phase is preferably 50% by mass or less, more preferably 45% by mass or less, and still more preferably 40% by mass or less, based on the total mass of the sputtering target from the viewpoint of increasing dispersibility with the other raw materials during mixing.

[0031]

(4. Third Element)

The ferromagnetic material sputtering target according to the present invention may contain, as a third element, a total of 30 mol% or less, for example 0.01 to 20 mol%, and typically 0.05 to 10 mol%, of one or two or more selected from the group consisting of B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Al, Si, and Zn as a

single metal or as an alloy. These elements are optionally added to improve characteristics as a magnetic recording medium. The blending ratio can be variously adjusted within the above-mentioned range, and any of the elements can maintain characteristics as an effective magnetic recording medium. It is noted that in the present invention, B is also treated as a metal.

[0032]

The third element may be present in the Co-Pt alloy particle phase or may be present in the Co particle phase. The third element may also be present in another particle phase distinguishable from the Co-Pt alloy particle phase and the Co particle phase. Such a particle phase can be present dispersed in the target structure. However, for the reason that finely dispersed the third element is more beneficial for reducing particle matter, it is preferable for the third element to be present in the Co-Pt alloy particle phase or in another fine particle phase. It is noted that if a third element forms not a particle phase of a single metal or an alloy, but a particle phase as an oxide, a nitride, a carbide, or a carbonitride, then such a particle phase is not treated as a particle phase of the third element, but rather is treated as a particle phase of a non-magnetic material (described below).

[0033]

When the third element is present in the other particle phase, it is preferable for the other particle phase to form a composite phase in which the other particle phase and the Co-Pt alloy particle phase are mutually dispersed in each other. Further, when a particle phase of a non-magnetic material (described below) is also present, it is preferable for the other particle phase to form a composite phase in which the other particle phase, the Co-Pt alloy particle phase, and the particle phase of the non-magnetic material are mutually dispersed in each other. When the third element forms the other particle phase, it is preferable for the particle phase of the third element to have an average particle size of 20 μm or less, more preferably 10

μm or less, and still more preferably 5 μm or less. However, if the average particle size of the particle phase of the third element is too small, oxidation may proceed during the mixed powder preparation step due to the oxygen in the atmosphere, and it is therefore preferable for the average particle size to be 0.5 μm or more, more preferably 1 μm or more, and still more preferably 2 μm or more.

[0034]

(5. Non-magnetic Material)

The ferromagnetic material sputtering target according to the present invention may contain a total of 25 mol% or less, for example 5 to 20 mol%, and typically from 5 to 15 mol%, of one or two or more non-magnetic materials selected from the group consisting of carbon, an oxide, a nitride, a carbide, and a carbonitride, as an added material. In this case, the sputtering target can have characteristics that are suitable for the material of a magnetic recording film having a granular structure, in particular a hard disk drive recording film employing a perpendicular magnetic recording system.

[0035]

Examples of carbides include carbides of one or two or more elements selected from the group consisting of B, Ca, Nb, Si, Ta, Ti, W, and Zr. Examples of oxides include oxides of one or two or more elements selected from the group consisting of Si, Al, B, Ba, Be, Co, Ca, Ce, Cr, Dy, Er, Eu, Ga, Gd, Ho, Li, Mg, Mn, Nb, Nd, Pr, Sc, Sm, Sr, Ta, Tb, Ti, V, Y, Zn, and Zr. Among oxides, it is preferable to add SiO₂ since it has a large effect of contributing to increasing the density of the sputtering target. Examples of nitrides include nitrides of one or two or more elements selected from the group consisting of Al, Ca, Nb, Si, Ta, Ti, and Zr. These non-magnetic materials may be appropriately added in accordance with the required magnetic characteristics of the magnetic thin film. It is noted that Cr oxide

and Co oxide are recognized as being different from the Cr and Co that are added as metals.

[0036]

The non-magnetic material can be present dispersed in the target structure as a non-magnetic material particle phase distinguishable from the Co-Pt alloy particle phase and the Co particle phase. In that case, it is preferable for the particle phase of the non-magnetic material to have an average particle size of 2 μm or less, more preferably 1 μm or less, and still more preferably 0.5 μm or less. However, if the average particle size of the particle phase of the non-magnetic material is too small, the particles may agglomerate with each other during the mixed powder preparation step and form clumps (coarse secondary particles), and therefore it is preferable for the average particle size to be 0.05 μm or more, more preferably 0.1 μm or more, and still more preferably 0.2 μm or more.

[0037]

(6. XRD Profile)

In one embodiment, the ferromagnetic material sputtering target according to the present invention has peaks at positions $2\theta = 33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$, respectively, in XRD measurement with a 2θ measurement range of 30° to 60° . By having such an XRD profile, it is possible for the sputtering target to have a larger flux leakage. Though it is not intended to limit the present invention by any theory, each of the above-mentioned peaks is derived from the $L1_0$ structure, and hence in view of the composition of the sputtering target according to the present invention, it is inferred that such peaks would be observed when the Co-Pt alloy particle phase forms an ordered phase having an $L1_0$ structure.

[0038]

Having peaks at positions $2\theta = 33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$, respectively, means that the ratio of the peak intensity to the

background intensity at each positions (average value of the intensity at $2\theta = 37.0$ to 38.0°) is 2 or more, and in general the ratio can be from 2 to 200.

[0039]

In particular, in order to increase the flux leakage, it is necessary for the $L1_0$ structure to be properly formed. For that reason, it is preferable for the strongest peak for the 2θ measurement range of 30° to 60° in the XRD measurement is $41.52 \pm 2^\circ$. $41.52 \pm 2^\circ$ is the strongest peak of the $L1_0$ structure of Co-Pt.

[0040]

In the present invention, the XRD measurement of the sputtering target is carried out under the following conditions. Using an X-ray diffractometer (in the Examples, an Ultima IV manufactured by Rigaku Corporation) as a measuring apparatus, Cu is used as the tube, and the measurement conditions are a tube voltage of 40 kv, a tube current of 30 mA, a scan speed of $4^\circ/\text{min}$, and a step of 0.02° . The measurement is carried out with respect to a plane horizontal to the sputtering surface by a $\theta/2\theta$ method. As long as the face is horizontal to the sputtering surface, the face may be the surface or a cut face.

[0041]

(7. Preparation method)

The ferromagnetic material sputtering target according to the present invention can be produced, for example, by the following procedure using a powder sintering method.

[0042]

A Co powder having a purity of 90 mol% or more, preferably 95 mol% or more, and more preferably 99.9 mol% or more, is provided. The Co powder may be prepared by grinding an ingot of melt-cast metal cobalt, or may be prepared by a gas atomizing method. The Co powder preferably has a median size of $30\ \mu\text{m}$ or more, more preferably $50\ \mu\text{m}$ or more, and still more preferably $100\ \mu\text{m}$ or more.

However, if the median size of the Co powder is too large, it may be difficult to uniformly mix with the other powder materials, or particle matter can be caused to form during sputtering. Therefore, the median size is preferably 300 μm or less, more preferably 200 μm or less, and still more preferably 150 μm or less. The median size can be adjusted by grinding or sieving.

[0043]

Further, a Co-Pt alloy powder is provided containing, under a condition of a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$), a total of metal Co and metal Pt of 70 mol% or more, preferably 80 mol% or more, more preferably 90 mol% or more, and still more preferably 99 mol% or more. From the viewpoint of developing the $L1_0$ structure, the Co-Pt alloy powder preferably has a median size of 7 μm or less, more preferably 6 μm or less, and still more preferably 5 μm or less. However, if the median size of the Co-Pt alloy powder is too small, oxidation may proceed during the mixing step due to the oxygen in the atmosphere, and it is therefore preferable for the median size to be 0.1 μm or more, more preferably 0.5 μm or more, and still more preferably 1 μm or more.

[0044]

Examples of a method for preparing such a Co-Pt alloy powder include firing a mixed powder of a Co powder and a Pt powder in a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$) at a heating temperature of 800°C to 1000°C under an inert atmosphere. According to this firing method, in XRD measurement in which the measurement range of 2θ is 30° to 60°, a sponge of a Co-Pt alloy having peaks at positions $2\theta = 33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$, respectively, can be obtained. Examples of the inert atmosphere include a vacuum atmosphere, a rare gas atmosphere such as an Ar gas atmosphere, and a nitrogen gas atmosphere. The median size can be adjusted by sieving a powder obtained by grinding the obtained sponge.

[0045]

Next, the Co-Pt alloy powder and the Co powder are mixed to obtain a mixed powder containing a total of 70 mol% or more of the metal Co and the metal Pt in a molar ratio of Co:Pt = X:100 - X ($59 \leq X < 100$).

[0046]

A powder of one or two or more third elements selected from the group consisting of B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Al, Si, and Zn is provided as needed. It is preferable to add the powder of the third element such that its total concentration in the mixed powder is the above-mentioned predetermined concentration in the sputtering target. The powder of the third element preferably has a median size of 20 μm or less, more preferably 10 μm or less, and still more preferably 5 μm or less. However, if the median size of the powder of the third element is too small, oxidation may proceed during the mixed powder preparation step due to the oxygen in the atmosphere, and it is therefore preferable for the median size to be 0.5 μm or more, more preferably 1 μm or more, and still more preferably 2 μm or more.

[0047]

A powder of one or two or more non-magnetic materials selected from the group consisting of carbon, an oxide, a nitride, a carbide, and a carbonitride is provided as needed. It is preferable to add the powder of the non-magnetic material such that its total concentration in the mixed powder is the above-mentioned predetermined concentration in the sputtering target. The powder of the non-magnetic material preferably has a median size of 2 μm or less, more preferably 1 μm or less, and still more preferably 0.5 μm or less. However, if the median size of the powder of the non-magnetic material is too small, oxidation may proceed during the mixed powder preparation step due to the oxygen in the

atmosphere, and it is therefore preferable for the median size to be 0.05 μm or more, more preferably 0.1 μm or more, and still more preferably 0.2 μm or more.

[0048]

A Cr powder is further provided as needed. In the case of adding a Cr powder, care should be taken so that the total concentration of metal Cr in the mixed powder is within the above-mentioned predetermined concentration range in the sputtering target. The Cr powder preferably has a median size of 10 μm or less, more preferably 5 μm or less, and still more preferably 3 μm or less. However, if the median size of the Cr powder is too small, oxidation may proceed during the mixed powder preparation step due to the oxygen in the atmosphere, and it is therefore preferable for the median size to be 1 μm or more, more preferably 1.5 μm or more, and still more preferably 2 μm or more.

[0049]

It is noted that the median size of each of the above-mentioned raw material powders refers to the particle size at an integral value of 50% (D50) based on the volume value in a particle size distribution obtained by a laser diffraction/scattering method. In the Examples, a model LA-920 particle size distribution measuring apparatus manufactured by HORIBA, Ltd. was used. The powder was dispersed in a solvent of ethanol and measured by a wet method. As the refractive index, the value of metal cobalt was used.

[0050]

The above-mentioned raw material powders were weighed so as to obtain a desired composition, and mixed together while grinding by a known technique such as a ball mill. At this time, it is desirable to fill the grinding vessel with an inert gas to suppress the oxidation of the raw material powder as much as possible. Examples of the inert gas include Ar and N_2 gas.

[0051]

The resultant mixed powder is molded and sintered in a vacuum atmosphere or an inert gas atmosphere by a hot-pressing method. In addition to a hot-pressing method, various other pressure sintering methods can also be used, such as a plasma discharge sintering method. In particular, a hot isostatic pressing method (HIP) is effective in improving the density of the sintered compact. From the viewpoint of improving the density of the sintered compact, it is preferable to carry out the hot-pressing method and the hot isostatic pressing method in this order.

[0052]

The retention temperature during sintering is preferably set to the lowest temperature in the temperature range at which the target becomes sufficiently dense. Although the temperature depends on the composition of the target, in most cases the temperature may be kept in a temperature range of 700 to 1200°C. Even within this temperature range, from the viewpoint of increasing the ordered phase of the $L1_0$ structure, the retention temperature during sintering is preferably set to 1050°C or less, more preferably 1000°C or less, and still more preferably 950°C or less. Further, the pressure during sintering is preferably set to 300 to 500 kg/cm². Although the retention temperature during hot isostatic pressing depends on the composition of the sintered compact, in most cases it is in a temperature range of 700 to 1200°C. Even within this temperature range, from the viewpoint of increasing the ordered phase of the $L1_0$ structure, it is preferably 1050°C or less, more preferably 1000°C or less, and still more preferably 950°C or less. In addition, it is preferable to set the applied pressure to 100 MPa or more.

[0053]

The sintering time is preferably 0.3 hours or more, more preferably 0.5 hours or more, and still more preferably 1.0 hour or more, in order to improve the density of sintered compact. The sintering time is preferably 3.0 hours or less, more

preferably 2.0 hours or less, and still more preferably 1.5 hours or less, in order to prevent coarsening of the crystal grains.

[0054]

The sputtering target according to the present invention can be produced by molding the obtained sintered compact into a desired shape by using a lathe or the like. The shape of the target is not particularly limited, and may be, for example, a flat plate shape (including a disk shape and a rectangular plate shape) and a cylindrical shape. The sputtering target according to the present invention is particularly useful as a sputtering target to be used for forming a magnetic recording film.

EXAMPLES

[0055]

Examples of the present invention are now shown below together with comparative examples, but these Examples are provided for a better understanding of the present invention and its advantages, and are not intended to limit the present invention.

[0056]

(1. Production of Sputtering Target)

As raw material powders, a Co powder, a Pt powder, a Co-Pt alloy powder, a Cr powder, a B_2O_3 powder, a TiO_2 powder, a SiO_2 powder, a Co_3O_4 powder, a Cr_2O_3 powder, a B powder, a Ru powder, a Ta Powder, a Ta_2O_5 powder, a CoO powder, a Si_3N_4 powder, and a SiC powder, each having the median size shown in Table 1, were provided. All of the raw material powders were a high purity product, and did not include other components other than inevitable impurities. The median size of these powders was adjusted appropriately by sieving.

[0057]

Of the above-mentioned raw material powders, the Co-Pt alloy powder was prepared by the following procedure. A Co powder having a median size of 3 μm and a Pt powder having a median size of 2 μm were rotated and mixed for 2 hours using an automatic mortar so that the Co molar ratio (Y) in the Co-Pt alloy powder would be the value shown in Table 3. Next, the obtained mixed powder was fired for 2 hours in a vacuum atmosphere as the pre-heat treatment temperature shown in Table 5 according to the test number. Then, the obtained sponge was ground, and obtained powder was sieved, to obtain a Co-Pt alloy powder having the respective median size shown in Table 1. However, as the Co-Pt powder in Comparative Example 3, powder obtained by sieving powder prepared by a gas atomization method was used.

[0058]

[Table 1]

[0059]

Next, each of the above-mentioned raw material powders was sealed in a ball mill pot having a capacity of 10 liters together with zirconia balls as grinding media in the mass ratio shown in Table 2 and rotated and mixed for 20 hours so that the molar ratio listed in the composition column of Table 3 was obtained according to the test number. Then, the obtained mixed powder was filled in a mold made of carbon and hot-pressed in a vacuum atmosphere at the temperature shown in Table 5 for a retention time of 2 hours and a pressure of 30 MPa to obtain sintered body. Next, the sintered body was removed from the hot press and subjected to hot isostatic pressing at the temperature shown in Table 5. Further, the applied pressure was set in the range of 100 to 200 MPa. The pressed sintered compact was then ground using a general-purpose lathe and a surface grinding machine to obtain a disk-shaped sputtering target having a diameter of 180 mm and a thickness of 5 mm. The target composition of each test number calculated from the mixing ratio of the raw material powders and the molar ratio (X) (%) of the metal Co to the total of the metal Co and the metal Pt in the target are shown in Table 3. Further, the molar ratio (Y) (%) (equivalent to the composition of the Co-Pt alloy powder) of metal Co to the total of metal Co and metal Pt in the Co-Pt alloy particle phase is shown in Table 3.

[0061]

[Table 3]

Test No.	Composition (mol%)	Molar Ratio of Co (X) ^{*1}	Molar Ratio of Co (Y) ^{*2}
Comparative Example 1	62Co-7Cr-20Pt-2B ₂ O ₃ -3TiO ₂ -3SiO ₂ -3Co ₃ O ₄	75.6	-
Comparative Example 2	68.5Co-19.7Pt-2B ₂ O ₃ -3TiO ₂ -3SiO ₂ -3.5Cr ₂ O ₃ -0.3Co ₃ O ₄	77.7	-
Comparative Example 3	88(80Co-5Cr-15Pt)-5CoO-7SiO ₂	84.3	50
Example 1	62Co-7Cr-20Pt-2B ₂ O ₃ -3TiO ₂ -3SiO ₂ -3Co ₃ O ₄	75.6	49
Example 2	68.5Co-19.7Pt-2B ₂ O ₃ -3TiO ₂ -3SiO ₂ -3.5Cr ₂ O ₃ -0.3Co ₃ O ₄	77.7	49
Example 3	64.4Co-5.4Cr-20.3Pt-2B ₂ O ₃ -3TiO ₂ -3SiO ₂ -2Co ₃ O ₄	76	38.8
Example 4	69.4Co-20Pt-2B ₂ O ₃ -3TiO ₂ -3SiO ₂ -2.6Cr ₂ O ₃	77.6	38.8
Example 5	61Co-9.5Cr-21Pt-2B ₂ O ₃ -2TiO ₂ -2SiO ₂ -2.5Co ₃ O ₄	74.3	28.1
Example 6	67Co-3Cr-21Pt-2B ₂ O ₃ -2TiO ₂ -2SiO ₂ -3Cr ₂ O ₃	76.1	28.1
Example 7	62.5Co-9Cr-21Pt-2B ₂ O ₃ -2TiO ₂ -2SiO ₂ -1.5Co ₃ O ₄	74.9	43.8
Example 8	66.2Co-4Cr-21Pt-2.3B ₂ O ₃ -2TiO ₂ -2SiO ₂ -2.5Cr ₂ O ₃	75.9	43.8
Example 9	68Co-19Pt-2TiO ₂ -6SiO ₂ -6CoO	78.6	50
Example 10	61Co-4Cr-14Pt-4B-4Ru-4Ta-3TiO ₂ -3SiO ₂ -3Cr ₂ O ₃	81.3	49
Example 11	59.5 Co -19.5Cr-10Pt-1Ta ₂ O ₅ -5TiO ₂ -5SiO ₂	85.6	49
Example 12	60Co-10Cr-9Pt-9Ru-5TiO ₂ -7CoO	86.9	48
Example 13	63Co-7Cr-20Pt-1Si ₃ N ₄ -1SiC-5TiO ₂ -3Cr ₂ O ₃	75.9	47
Example 14	50Co-20Pt-15TiO ₂ -15CoO	71.4	49
Example 15	85Co-10Pt-5SiO ₂	89.4	60

*1: Molar ratio of metal Co to the total of metal Co and metal Pt in target (%)

*2: Molar ratio of metal Co to the total of metal Co and metal Pt in Co-Pt alloy powder (Co-Pt alloy particle phase) (%)

[0062]

(2. Analysis of Target Structure)

Each sputtering target obtained by the above procedure was observed by following procedure to determine the average particle size of the Co particle phase, the Co-Pt alloy particle phase, the particle phase of the third element, and the non-magnetic material phase. It is noted that the Co particle phase, the Co-Pt alloy

particle phase, the particle phase of the third element, and the non-magnetic material phase were identified using FE-EPMA elemental mapping images. The results are shown in Table 4.

[0063]

(2-1. Co-Pt Alloy Particle Phase)

In the present invention, the average particle size of the Co-Pt alloy particle phase is calculated by the following method. A SEM photograph $70\ \mu\text{m}$ in length $\times$ $95\ \mu\text{m}$ in width is obtained by photographing at a magnification of 3000 a mirror-polished cut face of a face horizontal to the sputtering surface of the sputtering target. The photograph is divided into three sections at equal intervals in the length direction by two cutting lines horizontal to the width direction. The cut length of the Co-Pt alloy particle phase cut by each cutting line is measured, and the average value (μm) of the cut length is obtained for each field of view. Note that the thickness of the cutting line is taken to be 1/400th the length in the length direction of the photograph. This procedure is carried out for arbitrary 10 fields of view, and the average value of 10 fields of view is taken as the measured value. Also note that the Co-Pt alloy particle phase, which is only partly included the field of view, is excluded from the object to be measured.

[0064]

(2-2. Co Particle Phase)

In the present invention, the average particle size of the Co particle phase is calculated by the following method. Measurement of the size of the Co particle phase is carried out using a SEM photograph $1120\ \mu\text{m}$ in length $\times$ $1500\ \mu\text{m}$ in width obtained by photographing at a magnification of 220 a mirror-polished cut face of a face horizontal to the sputtering surface of the sputtering target. The photograph is divided into three sections at equal intervals in the length direction by two cutting lines horizontal to the width direction. The cut length of the Co particle phase cut

by each cutting line is measured, and the average value (μm) of the cut length is obtained for each field of view. Note that the thickness of the cutting line is taken to be 1/400th the length in the length direction of the photograph. This procedure is carried out for arbitrary 10 fields of view, and the average value of 10 fields of view is taken as the measured value. Also note that the Co particle phase, which is only partly included the field of view, is excluded from the object of what is to be measured.

[0065]

(2-3. Particle Phase of Third Element)

In the present invention, the average particle size of the particle phase of the third element when the third element forms an independent particle phase is calculated by the following method. A SEM photograph $215\ \mu\text{m}$ in length $\times$ $290\ \mu\text{m}$ in width is obtained by photographing at a magnification of 1000 a mirror-polished cut face of a face horizontal to the sputtering surface of the sputtering target. The photograph is divided into three sections at equal intervals in the length direction by two cutting lines horizontal to the width direction. The cut length of the particle phase of the third element cut by each cutting line is measured, and the average value (μm) of the cut length is obtained for each field of view. Note that the thickness of the cutting line is taken to be 1/400th the length in the length direction of the photograph. This procedure is carried out for arbitrary 10 fields of view, and the average value of 10 fields of view is taken as the measured value. Also note that the particle phase of the third element, which is only partly included the field of view, is excluded from the object of what is to be measured.

[0066]

(2-4. Particle Phase of Non-Magnetic Material)

In the present invention, the average particle size of the particle phase of the non-magnetic material is calculated by the following method. A SEM photograph

70 μm in length $\times$ 95 μm in width is obtained by photographing at a magnification of 3000 a mirror-polished cut face of a face horizontal to the sputtering surface of the sputtering target. The photograph is divided into three sections at equal intervals in the length direction by two cutting lines horizontal to the width direction. The cut length of the particle phase of the non-magnetic material cut by each cutting line is measured, and the average value (μm) of the cut length is obtained for each field of view. Note that the thickness of the cutting line is taken to be 1/400th the length in the length direction of the photograph. This procedure is carried out for arbitrary 10 fields of view, and the average value of 10 fields of view is taken as the measured value. Also note that the particle phase of the non-magnetic material, which is only partly included the field of view, is excluded from the object of what is to be measured.

[0067]

[Table 4]

Test No.	Average Particle Size in Target Structure (μm)			
	Co particle phase (coarse powder)	Co-Pt alloy particle phase	Third element particle phase	Non-magnetic material particle phase
Comparative Example 1	41	-	-	0.4
Comparative Example 2	30	-	-	0.5
Comparative Example 3	82	75	-	1
Example 1	43	2.5	-	0.4
Example 2	32	2	-	0.5
Example 3	42	2.4	-	0.5
Example 4	45	2.5	-	0.5
Example 5	43	2	-	0.4
Example 6	47	2.6	-	0.5
Example 7	38	2.3	-	0.5
Example 8	40	2.4	-	0.4
Example 9	121	4.2	-	1.1
Example 10	119	5.8	15	1.3
Example 11	252	2.6	-	2
Example 12	58	3.5	5	1.8
Example 13	59	1.8	-	1.5
Example 14	296	0.6	-	2
Example 15	61	1.9	-	1.2

[0068]

(3. Measurement of Flux leakage)

For each sputtering target obtained by the above procedure, the flux leakage was measured in conformity to ASTM F 2086-01 (Standard Test Method for Pass Through Flux of Circular Magnetic Sputtering Targets, Method 2). The center of the target was fixed, and the flux leakage density (PTF) was measured by rotating the target by 0°, 30°, 60°, 90°, and 120°. The measured flux leakage density was divided by the value of the reference field defined by ASTM, and then multiplied by 100 to express as a percentage. The results averaged over those five points are listed in Table 5 as the average flux leakage density (PTF (%)).

[0069]

(4. Measurement of Particle Matter)

Each sputtering target obtained by the above procedure was mounted to a magnetron sputtering apparatus (C-3010 sputtering system manufactured by CANON ANELVA CORPORATION), and sputtering was performed. The conditions for sputtering were an input power of 1 kW and an Ar gas pressure of 1.7 Pa. After pre-sputtering at 2 kWhr, film deposition was carried out for 20 seconds on a 4-inch diameter silicon substrate. Then, the number of particles having a particle size of 0.07 μm or more adhering to the substrate was measured with a surface foreign matter inspection apparatus (Candela CS 920, manufactured by KLA-Tencor). The results are shown in Table 5.

[0070]

(5. XRD Measurement)

XRD Measurement was carried out under the above-mentioned conditions by using an X-ray diffractometer (Ultima IV manufactured by Rigaku) on a mirror-polished cut face of a face horizontal to the sputtering surface of each sputtering target obtained by the above procedure. In the XRD measurement, the 2θ measurement range was set to 30° to 60° , and the ratio of the peak intensity at the respective diffraction angles (2θ) of $33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$ to the background intensity (average value of the intensity at $2\theta = 37.0^\circ$ to 38.0°) was investigated. The results are shown in Table 5. Further, the XRD profiles of Comparative Example 1, Comparative Example 2, Comparative Example 3, Example 1, Example 2, Example 3, Example 5, and Example 7 are shown in Figures 1 to 8, respectively.

[0071]

[Table 5]

Test No.	Pre-heat treatment temperature of Co-Pt (°C)	Hot-pressing temperature (°C)	Hot isostatic pressing temperature (°C)	Flux leakage (PTF)	Particles (number)	Peak Intensity (ratio versus background) At Each Diffraction Angle) (2θ)				
						33.27±2°	41.52±2°	47.76±2°	49.44±2°	54.21±2°
Comparative Example 1	none	900	900	28.8	222	1	3.7	2.7	19	1
Comparative Example 2	none	900	900	28.5	210	1.3	1.7	1	4.2	1
Comparative Example 3	none	1100	-	44.2	205	1	5.7	1.5	5.3	1
Example 1	900	900	900	48.2	94	5.6	26	6.7	7.3	3.8
Example 2	900	900	900	49.5	106	7.7	42	9	8.7	4.3
Example 3	900	900	900	29.9	107	4.7	19.3	10	6	2.9
Example 4	900	900	900	30.5	105	6.3	28.7	14	7.7	4
Example 5	1000	900	900	61.8	82	20.2	77.5	20.7	12.7	8.9
Example 6	1000	900	900	62.1	97	7.3	57	12.3	10.3	4.3
Example 7	800	900	900	58.9	98	13.8	59	20.7	13.3	7.1
Example 8	800	900	900	56.8	85	20	80.7	24.3	16	11
Example 9	900	1000	1000	40.5	116	4	29.3	8	7.3	5
Example 10	900	800	800	57.3	133	4	23.3	14.7	7	2.7
Example 11	900	1000	1000	65	90	4.7	37	7	11.3	4
Example 12	900	1000	1000	47.9	111	5.7	41.7	13.7	9.3	5.7
Example 13	900	900	900	48.3	128	6.7	30.3	15.3	9	3.7
Example 14	900	1000	1000	62.5	149	6.3	50.3	12.3	10.3	4.7
Example 15	900	1050	1000	33.7	104	3.7	33.3	11.3	12	2.3

What is claimed is:

1. A ferromagnetic material sputtering target comprising a total of 70 mol% or more of metal Co and metal Pt in a molar ratio of Co:Pt = X:100 - X ($59 \leq X < 100$) and comprising 0 mol% or more and 20 mol% or less of metal Cr,
wherein the ferromagnetic material sputtering target comprises:
a Co particle phase comprising 90 mol% or more of metal Co and having an average particle size of 30 to 300 μm ; and
a Co-Pt alloy particle phase that, under a condition of a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$), comprises a total of 70 mol% or more of metal Co and metal Pt and has an average particle size of 7 μm or less.
2. The ferromagnetic material sputtering target according to claim 1, wherein the ferromagnetic material sputtering target comprises a total of 30 mol% or less of one or two or more third elements selected from a group consisting of B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Al, Si, and Zn.
3. The ferromagnetic material sputtering target according to claim 1 or 2, wherein the ferromagnetic material sputtering target comprises a total of 25 mol% or less of one or two or more non-magnetic materials selected from a group consisting of carbon, an oxide, a nitride, a carbide, and a carbonitride.
4. The ferromagnetic material sputtering target according to any one of claims 1 to 3, wherein the ferromagnetic material sputtering target has peaks at positions $2\theta = 33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$, respectively, in XRD measurement with a 2θ measurement range of 30° to 60° .
5. A method of producing the ferromagnetic material sputtering target according to any one of claims 1 to 4, wherein the method comprises:
a step of providing a Co powder comprising 90 mol% or more of metal Co and having a median size of 30 to 300 μm ;

a step of providing a Co-Pt alloy powder that, under a condition of a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$), comprises a total of 70 mol% or more of metal Co and metal Pt and has a median size of 7 μm or less;

a step of obtaining a mixed powder by mixing the Co-Pt alloy powder and the Co powder such that, under a condition of a molar ratio of Co:Pt = X:100 - X ($59 \leq X < 100$), the mixed powder comprises a total of 70 mol% or more of metal Co and metal Pt and comprises 0 mol% or more and 20 mol% or less of metal Cr; and

a step of firing the mixed powder.

6. The method according to claim 5, wherein the step of obtaining the mixed powder further comprises mixing one or two or more powders selected from a group consisting of B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Al, Si, and Zn.

7. The method according to claim 5 or 6, wherein the step of obtaining the mixed powder further comprises mixing one or two or more non-magnetic materials selected from a group consisting of carbon, an oxide, a nitride, a carbide, and a carbonitride.

8. The method according to any one of claims 5 to 7, wherein the step of providing the Co-Pt alloy powder that has the median size of 7 μm or less comprises:

a step of obtaining a first sintered compact by firing a mixed powder of a Co powder and a Pt powder in a molar ratio of Co:Pt = Y:100 - Y ($20 \leq Y \leq 60.5$) at a heating temperature of 800°C to 1000°C such that the first sintered compact has peaks at positions $2\theta = 33.27 \pm 2^\circ$, $41.52 \pm 2^\circ$, $47.76 \pm 2^\circ$, $49.44 \pm 2^\circ$, and $54.21 \pm 2^\circ$, respectively, in XRD measurement with a 2θ measurement range of 30° to 60°; and

a step of grinding the first sintered compact.

9. A method of producing a magnetic recording film comprising using the sputtering target according to any one of claims 1 to 4.