

SPUTTERING TARGET CONTAINING Co OR Fe

[TECHNICAL FILED]

[0001] The present invention relates to a magnetic material sputtering target
5 used for depositing a granular film on a magnetic material thin film of a magnetic
recording medium, in particular, on a magnetic recording medium of a hard disk
adopting the perpendicular magnetic recording system, and also relates to a
nonmagnetic-grain dispersed magnetic material sputtering target comprising Co
or Fe as a main component capable of controlling an abnormal discharge due to
10 a nonmagnetic material which may cause particle generation during sputtering.

BACKGROUND

[0002] Materials based on Co, Fe and Ni as ferromagnetic metals are used for a
15 recording layer of a hard disk adapting the perpendicular magnetic recording
system. Among these, a composite material made from a Co-Cr based, a
Co-Pt based, a Co-Cr-Pt based, a Fe-Pt based ferromagnetic alloy having Co or
Fe as a main component and a nonmagnetic inorganic material are used. And,
these magnetic thin films for magnetic recording media of hard disks are often
20 produced by sputtering a ferromagnetic material sputtering target made from
the foregoing materials in light of its high productivity.

[0003] As a method of manufacturing such a sputtering target for a magnetic
recording medium, the dissolution method and the powder metallurgy method
may be considered. Which method may be used depends on required
25 properties and cannot be generalized. Nonetheless, a sputtering target
comprising a ferromagnetic alloy and nonmagnetic inorganic grains, which is
used for a recording layer of a hard disk of the perpendicular magnetic recording
system, is generally manufactured by the powder metallurgy method. This is
because manufacturing is difficult by the dissolution method since the inorganic
30 grains need to be uniformly dispersed in an alloy base.

[0004] In the case of the powder metallurgy method, for example, Patent
Document 1 proposes a method of obtaining a sputtering target for a magnetic
recording medium in Patent Document 2, comprising: mixing a powder mixture
obtained by mixing Co powder, Cr powder, TiO₂ powder and SiO₂ powder having
35 a Co spherical powder using a planetary mixer, and molding the resulting mixed
powder by hot press.

The target structure in this case appears to have a spherical phase (B) in

a phase (A) which is a base metal where inorganic grains are uniformly dispersed (see Fig. 1 in Patent Document 2). The structure as described above is good in terms of improving leakage magnetic flux. However, it cannot be considered as a suitable sputtering target for a magnetic recording medium in terms of suppressing particle generation during sputtering.

[0005] Patent Document 2 proposes a method of obtaining a sputtering target for depositing a thin film of a magnetic recording medium, comprising: mixing a Co-Cr binary alloy powder, a Pt powder and a SiO₂ powder, and hot pressing the resulting mixed powder.

It describes that a Pt phase, a SiO₂ phase and a Co-Cr binary alloy phase were observed in the target structure in this case although not shown in the figures, and a dispersed phase was observed around the Co-Cr binary alloy phase. It cannot be said that the structure as described above is a suitable sputtering target for a magnetic recording medium.

[0006] Further, Patent Document 3 proposes a sputtering target comprising a matrix phase of Co, Pt and metal oxide phases having an average grain diameter of 0.05 μm or more and less than 7.0 μm, and also proposes to obtain the target where deposition efficiency can be improved by suppressing the growth of crystal grain to achieve a low magnetic permeability and a high density

In addition, Patent Document 4 describes that the average grain diameter of grains formed in oxide phases is 3 μm or less. Patent Document 5 describes that silica grains or titania grains satisfy $2 \leq D_p / D_n$ wherein D_n represents a grain diameter in a direction perpendicular to a main surface of a sputtering target at a cross section perpendicular to the main surface of the target, and D_p represents a grain diameter in a direction parallel to the above main surface.

However, none of these conditions are sufficient, and a demand for further improvement still remains at present.

[Citation List]

[Patent Document]

[0007] Patent Document 1: WO2011/089760

Patent Document 2: Japanese Patent Application Laid-Open No. 2009-1860

Patent Document 3: Japanese Patent Application Laid-Open No. 2009-102707

Patent Document 4: Japanese Patent Application Laid-Open No. 2009-215617

Patent Document 5: Japanese Patent Application Laid-Open No. 2011-222086

Patent Document 6: Japanese Patent Application No. 2012-036562

SUMMARY OF INVENTION

[Technical Problem]

[0008] Generally, nonmagnetic materials contained such as SiO₂, Cr₂O₃, TiO₂ serve as insulators in nonmagnetic material grain-dispersed ferromagnetic sputtering targets having Co or Fe as a main component, and therefore, they may cause an abnormal discharge. This abnormal discharge, in turn, will pose a problem of particle generation during sputtering.

[0009] In view of the above problems, an object of the present invention is to suppress the aforementioned abnormal discharge due to nonmagnetic materials to reduce particle generation caused by an abnormal discharge during sputtering while maintaining high PTF. Though an attempt to reduce a probability of an abnormal discharge has been made by decreasing a grain size of a nonmagnetic material grain so far, an acceptable particle level is becoming stricter as the recording density of a magnetic recording medium is being improved. Accordingly, an object of the present invention is to provide a more improved nonmagnetic material grain-dispersed ferromagnetic sputtering target.

[Solution to Problem]

[0010] After intensive studies to solve the above problems, the present inventors discovered that by adjusting a structure (nonmagnetic material grains) of a target, a target with suppressed occurrence of an abnormal discharge due to a nonmagnetic material during sputtering and having less frequent particle generation can be obtained.

[0011] Based on these findings, the present invention provides the followings.

1) A sintered sputtering target made from a material having nonmagnetic material grains dispersed in a magnetic material containing Co or Fe, comprising:

a structure observed on a polished surface of the target comprises nonmagnetic material grains having an average grain diameter of 1.8 μm or less, a metal phase containing Co or Fe in which nonmagnetic material grains are dispersed and metal grains containing Co or Fe;

nonmagnetic material grains having a difference of 0.7 μm or less between a maximum diameter and a minimum diameter is 60% or more of nonmagnetic material grains in a structure observed on the polished surface of the target, defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of the nonmagnetic material grain and a minimum diameter as the smallest value of distances between two parallel

lines when the two lines sandwich the grain; and

one or more metal grains having a sum of the maximum diameter and the minimum diameter of 30 μm or more are present on average in 1 mm^2 field of view, defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of the metal grain, and a minimum diameter as the smallest value of distances between two parallel lines when the two lines sandwich the metal grain.

2) The sputtering target according to 1), wherein the nonmagnetic material grain is one or more oxides selected from B_2O_3 , CoO , Co_3O_4 , MnO , Mn_3O_4 , SiO_2 , SnO_2 , TiO_2 , Ti_2O_3 , Cr_2O_3 , Ta_2O_5 , WO_2 , WO_3 , ZrO_2 in an amount of 0.5 or more and 20 mol% or less.

3) The sputtering target according to 1) or 2), wherein Cr is contained at 0 mol% to 15 mol%, Pt is at 5 mol% or more and 30 mol% or less, the remainder being Co and unavoidable impurities except for the nonmagnetic material.

4) The sputtering target according to 3), further comprising one or more elements selected from Mg, Al, Si, Mn, Nb, Mo, Ru, Pd, Ta, W, B in an amount of 0.5 mol% or more and 12 mol% or less.

5) The sputtering target according to any one of 1) to 4), wherein the metal grains comprise Co or Fe.

[Effects of Invention]

According to the nonmagnetic material grain-dispersed magnetic material sputtering target of the present invention adjusted in this way, a target can be obtained showing no occurrence of abnormal discharge due to a nonmagnetic material during sputtering and having less frequent particle generation while maintaining high PTF. Advantageously, by this, a cost improving effect may be obtained from an improved yield.

BRIEF DESCRIPTION OF DRAWINGS

[0012] Fig. 1 shows an image (a photograph) illustrating the structure of a Co-Pt-Cr-SiO₂-TiO₂-Cr₂O₃ target from Example 1.

Fig. 2 shows an image (an enlarged photograph of Fig. 1) illustrating the structure of the target from Example 1 in which nonmagnetic material grains are dispersed in a metallic phase.

Fig. 3 shows an image obtained by subjecting Fig. 2 to an image analysis processing (binary processing) so that a nonmagnetic material grain is well

defined.

Fig. 4 shows an image (a photograph) illustrating the structure of a Co-Pt-Ru-Ta-SiO₂-TiO₂-CoO-B₂O₃ target from Example 2.

Fig. 5 shows an image (an enlarged photograph of Fig. 4) illustrating the structure of the target from Example 2 in which nonmagnetic material grains are dispersed in a metallic phase.

DESCRIPTION OF EMBODIMENTS

[0014] The sputtering target of the present invention is a sintered sputtering target made from a material in which nonmagnetic material grains are dispersed in a magnetic material containing Co or Fe, wherein a structure observed on a polished surface of the target comprises nonmagnetic material grains having an average grain diameter of 1.8 μm or less, a metal phase containing Co or Fe in which nonmagnetic material grains are dispersed and metal grains containing Co or Fe. When an average grain diameter of nonmagnetic material grains is set to 1.8 μm or less, particle generation can be suppressed

[0015] Previously, the present inventors found that the shape of a nonmagnetic material grain is preferably a perfect sphere, or at least a shape similar to that may be an effective means for preventing particle generation (Patent Document 6).

That is, a certain amount of an oxide (a nonmagnetic material) needs to be present in order to improve magnetic properties. However, when it is irregular in shape, the oxide may present in some places but not in others in a given area on the surface of a target, resulting in heterogeneous distribution and thus more frequent occurrence of segregation. If grains are complete or almost-complete spherical oxide grains, segregation is less significant because the grains are all similar in shape, and particle generation can be effectively controlled.

[0016] By the above findings of the present invention, in a case when defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of a nonmagnetic material grain observed on a polished surface of a target and a minimum diameter as the smallest value of distances between any two parallel lines when the two lines sandwich that grain, a maximum diameter differs from the minimum diameter by 0.7 μm or less.

Further, preferably, such nonmagnetic material grains occupy a majority of a target, i.e., 60% or more, more preferably 90% or more and even more preferably 100%. By this, particle generation can be significantly suppressed.

[0017] The present invention, which further provides new findings in addition to the above findings, can significantly suppress particle generation by specifying not only the shape of a nonmagnetic material grain but also the shape of a metal grain containing Co or Fe.

5 That is, in a case when a maximum diameter is defined as the greatest value of distances between any two points on the outer periphery of a metal grain observed on a polished surface of a target, and a minimum diameter as the smallest value of distances between any two parallel lines when the two lines sandwich the metal grain, the number of metal grains present in 1 mm² field of view having a sum of the maximum and the minimum diameter of 30 μm or more is one or more on average, preferably 3 or more on average, even more preferably 5 or more on average.

10 Note that in the present invention, microscopic observations are performed at any 5 positions within the surface of a target to count the number of metal grains present in 1 mm² field of view, having a sum of the maximum and the minimum diameter of 30 μm or more at each position, and then the average number is computed from the sum.

[0018] Defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of a metal grain, and a minimum diameter as the smallest value of distances between any two parallel lines when the two lines sandwich the metal grain, when one or more having a sum of the maximum and the minimum diameter of 30 μm or more are present on average in 1 mm² field of view, leakage magnetic flux is large. Then, when used in a magnetron sputtering system, the promotion of ionization of inert gas is efficiently promoted, and stable discharge can be obtained.

25 In contrast, defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of a metal grain, and a minimum diameter as the smallest value of distances between any two parallel lines when the two lines sandwich the metal grain, when the sum of the maximum and the minimum diameter is less than 30 μm, or less than one metal grain having a sum of the maximum and the minimum diameter of 30 μm or more are present on average in 1 mm² field of view, the above effect cannot practically be obtained.

30 Further, in a case where the sum of the maximum and the minimum diameters is 50 μm or more, the above effect can be more strongly obtained, but in a case where the sum of the maximum and the minimum diameters is 300 μm or more, the distribution of oxide particles may become heterogeneous.

[0019] The ferromagnetic sputtering target of the present invention is useful particularly for a Co based alloy such as a Co-Cr based alloy, a Co-Pt based alloy, a Co-Cr-Pt based alloy or an Fe based alloy such as an Fe-Pt based alloy. However, the present invention can also be applied to already known
5 ferromagnetic materials, and a composition ratio of components required as a magnetic recording medium may be appropriately adjusted depending on purposes.

In the case of a Co based alloy, a sputtering target may be obtained comprising 0 mol% to 15 mol% of Cr, 5 mol% to 30 mol% of Pt, the remainder
10 being Co and unavoidable impurities except for a nonmagnetic material. In the case of an Fe based alloy, a sputtering target may be obtained comprising more than 0 mol% and 60 mol% or less of Pt, and the remainder being Fe and unavoidable impurities except for a nonmagnetic material.

Note that these compositions show suitable numerical ranges for taking
15 advantage of properties as a ferromagnetic material, and of course, numerical values other than these may be used.

[0020] A nonmagnetic material added to the ferromagnetic material comprises one or more oxides selected from B_2O_3 , CoO, Co_3O_4 , MnO, Mn_2O_3 , SiO_2 , TiO_2 , Ti_2O_3 , Cr_2O_3 , Ta_2O_5 , WO_2 , WO_3 , ZrO_2 , which are usually contained in a target in
20 an amount of 0.5 or more and 20 mol% or less. Depending on intended types of ferromagnetic films, these oxides can be arbitrarily selected for addition. The loading amount described above is an effective amount to have an effect of addition.

One or more elements selected from Mg, Al, Si, Mn, Nb, Mo, Ru, Pd, Ta, W, B may also be added to the sputtering target of the present invention in an
25 amount of 0.5 to 12 mol%. These elements can be arbitrarily added to improve properties as a magnetic recording medium. The loading amount described above is an effective amount to have an effect of addition.

[0021] Further, the structure of the sputtering target of the present invention comprises nonmagnetic material grains, a metal phase containing Co or Fe in
30 which nonmagnetic material grains are dispersed and metal grains. The metal grains preferably comprise Co or Fe.

The above metal grains have a higher maximum permeability than the metal base having different compositions (the metal phase having nonmagnetic material grains dispersed therein), and show a structure in which each is
35 separated by a surrounding structure comprising the metal base. To date, the

mechanism of improved leakage magnetic field in a target having such a structure is not fully understood. Nonetheless, a state in which magnetic flux leaks outside appears to be energetically preferred because it has higher magnetostatic energy as compared to a structure having a uniform magnetic permeability when high dense regions and low dense regions in terms of magnetic flux occur within the target.

[0022] The sputtering target of the present invention may be manufactured by the powder metallurgy method. In the case of the powder metallurgy method, except for a metal coarse powder described below, a raw metal powder such as Co, Cr, Pt, Fe, and a nonmagnetic material raw powder such as SiO₂, and if needed, an additive metal powder such as Ru are prepared. As for a grain size of a raw material, an average grain diameter of 10 μm or less is preferably used for a metal powder, and an average grain diameter of 5 μm or less is preferably used for a nonmagnetic material powder. The fine structure of the present invention may be more easily achieved if a nonmagnetic material raw powder is as spherical as possible. Alternatively, an alloy powder of these metals may be prepared instead of a powder of each metal element. Note that the grain diameter of a powder may be measured with a laser diffraction particle size distribution analyzer (HORIBA LA-920).

[0023] Then, these metal powders and alloy powders are weighed out to give a desired composition and pulverized and mixed with well-known methods by using a ball mill or the like. In order to reduce mixing time to improve productivity, a high energy ball mill is preferably used. Here, for a metal raw material powder, a small amount of a metal coarse powder having at least one component with a grain diameter ranging from 50 μm to 300 μm is preferably mixed. In that case, addition is performed after mixing using a ball mill for a long time has finished. Otherwise, a mild mixing machine such as a mixer showing no grindability is preferably used for mixing in view of maintaining the grain diameter. Alternatively, addition may be performed in the middle of ball mill mixing, and then ball mill mixing may be performed for a short time. Thus, a metal grain becomes flat, and the shape will create a large difference between the major and the minor diameters.

[0024] A metal grain may be made into a spherical shape or a flat shape (a flake-like shape) in this way. A metal grain having a spherical shape or a flat shape has advantages and disadvantages depending on each shape. Preferably the shape is selected depending on the intended use of a target.

Specifically, in the case of a spherical shape, vacancy may be less prone

to occur at the interface between a metal base (A) and a phase (B) when manufacturing a target material by the sintering method, leading to an increased density of a target. Moreover, since a surface area is smaller if the volume is the same in the case of a spherical shape, the diffusion of a metal element between a metal base (A) and a phase (B) may not easily promoted when sintering the target material. Note that a spherical shape as used herein refers to a three-dimensional shape including a perfect sphere, a pseudo perfect sphere, an ellipse (a spheroid), a pseudo ellipse. In all cases, the difference between the major axis and the minor axis shall be 0 to 50%.

Meanwhile, a metal grain having a flat shape can advantageously prevent detachment of the metal grain from a surrounding metal base (A) during sputtering, literally by the wedge effect. Further, by destroying a spherical shape, the bias in an erosion rate which may easily be caused in the case of a spherical shape can be reduced, and particle generation due to a boundary of different erosion rates can be controlled.

[0025] In the present invention, it is important that when a maximum diameter is defined as the greatest value of distances between any two points on the outer periphery of a nonmagnetic material grain observed on a polished surface of a target, and a minimum diameter as the smallest value of distances between any two parallel lines when the two lines sandwich the grain, a maximum diameter differs from the minimum diameter by 0.7 μm or less.

Further, in the present invention, it is particularly important that when a maximum diameter is defined as the greatest value of distances between any two points on the outer periphery of a metal grain observed on a polished surface of a target, and a minimum diameter as the smallest value of distances between any two parallel lines when the two lines sandwich the metal grain, the number of metal grains present in 1 mm^2 field of view having a sum of the maximum and the minimum diameter of 30 μm or more is one or more on average.

The maximum and the minimum diameters are determined by displaying a microscopic image of a polished surface of a target on PC, and performing analysis with an image processing analysis software. A KEYENCE shape analysis software (VK-Analyzer VK-H1A1) was used for the image processing analysis software.

[0026] A mixed powder obtained as described above is sintered using a hot press or a hot isostatic press. Depending on a component composition of a target, if conditions in which nonmagnetic material grains becomes a perfect

sphere and conditions in which metal grains become a flat shape are found by selecting mixing conditions and sintering conditions of the above raw materials, and those manufacturing conditions are fixed, a sintered target in which the nonmagnetic material grains and the metal grains are dispersed therein can always be obtained.

EXAMPLES

[0027] The present invention is now explained with reference to Examples and Comparative Examples. Note that Examples are presented for merely illustrative purposes, and the present invention shall in no way be limited to these. That is, the present invention is only defined by the claims, and shall encompass various modifications other than those in Examples included in the present invention.

(Example 1)

[0028] Co powder having an average grain diameter of 4 μm , Cr powder having an average grain diameter of 5 μm , Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and TiO_2 powder having an average grain diameter of 1.2 μm , a spherical SiO_2 powder having an average grain diameter of 0.7 μm , Cr_2O_3 powder having an average grain diameter of 1 μm were prepared as nonmagnetic material powders. Further, Co coarse powder adjusted to have the grain diameter in the range of 50 μm to 150 μm was prepared, and the weight ratio of the Co powder having an average grain diameter of 4 μm to the Co coarse powder was 7:3. The powders were weighed out in this composition ratio to give 2000 g..

Composition: 69Co-18Pt-2Cr-5SiO₂-2TiO₂-4Cr₂O₃ (mol%)

[0029] The weighed powders, except for the Co coarse powder, were subsequently sealed in a 10-liter ball mill pot together with tungsten alloy balls as grinding media and rotated for 120 hours for mixing. Further, the Co coarse powder was added to the ball mill pot, and mixed for 1 hour. The obtained mixed powder was filled in a carbon mold, and under conditions of a vacuum atmosphere, a temperature of 1100°C, a holding time of 2 hours and a pressure of 30 MPa to obtain a sintered compact. This was further subjected to be cut with a lathe into a disk-shaped target with a diameter of 180 mm and a thickness of 5 mm.

[0030] The average leakage magnetic flux density was measured and it was 30%. Note that leakage magnetic flux was measured in accordance with ASTM

F2086-01 (Standard Test Method for Pass Through Flux of Circular Magnetic Sputtering Targets, Method 2). Specifically, the target was fixed at the center, and rotated for 0 degree, 30 degrees, 60 degrees, 90 degrees, 120 degrees to measure leakage magnetic flux densities. The resulting values were divided back by the value of the reference field defined in ASTM, and then multiplied by 100 to express them in percent. Then, the result from averaging these 5 points was taken as the average leakage magnetic flux density, i.e., 'pass through flux: PTF' (%).

[0031] Structural observation of a polished face of the target with a microscope showed that metal grains were scattered in a structure in which nonmagnetic material grains were dispersed in a metal phase as shown in figure 1. When defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of a metal grain, and a minimum diameter defining the smallest value of distances between any two parallel lines when the two lines sandwich the metal grain, the number of metal grains having a sum of the maximum and the minimum diameter of 30 μm or more in 1 mm^2 field of view was 40 on average.

Further, Fig. 2 shows an enlarged view of Fig. 1 to observe nonmagnetic material grains. When defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of a nonmagnetic material grain and a minimum diameter as the smallest value of distances between any two parallel lines when the two lines sandwich the grain, nonmagnetic material grains having a difference between the maximum and the minimum diameter of 0.7 μm or less account for 85% in 1 mm^2 field of view, and the average grain diameter was 0.75 μm .

Note that a microscopic image was displayed on a PC screen, and an image analysis processing (binary processing) was performed to obtain sharply defined oxide grains (black portions) as shown in Fig. 3, and then a maximum diameter, a minimum diameter, the average grain diameter of nonmagnetic material grains were computed.

[0032] The target was subsequently placed in DC magnetron sputtering device to perform sputtering. The sputtering conditions were a sputtering power of 1.2 kW, an Ar gas pressure of 1.5 Pa, and pre-sputtering was performed at 2 kWhr. Then sputtering was performed on a silicon substrate having a diameter of 4 inches at a target deposition thickness of 1000 nm. The number of particles adhering on the substrate was counted with a particle counter, and the number of particles on the silicon substrate was 4.

Even when spattering is not performed, the number of particles on the silicon substrate is occasionally counted to be 0 to 5 with a particle counter, and thus the number of 4 in this example can be said to be an extremely low level.

(Example 2)

[0033] Co powder having an average grain diameter of 4 μm , Pt powder having an average grain diameter of 3 μm , Ru powder having an average grain diameter of 7 μm , Ta powder having an average grain diameter of 6 μm were prepared as metal raw powders, and TiO_2 powder having an average grain diameter of 1.2 μm , a spherical SiO_2 powder having an average grain diameter of 0.7 μm , CoO powder having an average grain diameter of 0.8 μm , a B_2O_3 powder having an average grain diameter of 5 μm were prepared as oxide powders. Further, Co coarse powder adjusted to have the grain diameter in the range of 50 μm to 300 μm was prepared, and the weight ratio of the Co powder having an average grain diameter of 4 μm to the Co coarse powder was 7:3. The powders were weighed out in this composition ratio to give 2000 g.

Composition: 61.2Co-22Pt-3Ru-0.8Ta-6SiO₂-2TiO₂-4CoO-1B₂O₃ (mol%)

[0034] The weighed powders, except for the Co coarse powder, were subsequently sealed in a 10-liter ball mill pot together with tungsten alloy balls as grinding media and rotated for 120 hours for mixing. Then, the Co coarse powder was further added to the ball mill pot, and mixed for 1 hour. The obtained mixed powder was filled in a carbon mold, and hot pressed under conditions of a vacuum atmosphere, a temperature of 1000°C, a holding time of 2 hours and a pressure of 30 MPa to obtain a sintered compact. This was cut with a lathe into a disk-shaped target having a diameter of 180 mm and a thickness of 5 mm.

[0035] The average leakage magnetic flux density of the target in Example 2 was 28%. Structural observation of a polished face of the target with a microscope showed that metal grains were scattered in a structure in which nonmagnetic material grains were dispersed in a metal phase as shown in figure 4. The number of metal grains having a sum of the maximum and the minimum diameter of 30 μm or more in 1 mm^2 field of view was 19 on average as evaluated in Example 1. Further, an enlarged view of Fig. 4 is shown in Fig. 5 to observe nonmagnetic material grains. The ratio of nonmagnetic material grains having a difference between the maximum and the minimum diameter of 0.7 μm or less as evaluated in Example 1 was 64%, and the average grain diameter was 1.26 μm .

[0036] The target was subsequently placed in DC magnetron sputtering device to perform sputtering. The sputtering conditions were a sputtering power of 1.2 kW, an Ar gas pressure of 1.5 Pa, and pre-sputtering was performed at 2 kWhr as in Example 1. Then sputtering was performed on a silicon substrate having a diameter of 4 inches at a target deposition thickness of 1000 nm. The number of particles adhering on the substrate was counted with a particle counter, and the number of particles on the silicon substrate was 4.

(Example 3)

[0037] Co powder having an average grain diameter of 4 μm , Pt powder having an average grain diameter of 3 μm , Co-B powder having an average grain diameter of 7 μm were prepared as metal raw powders, and TiO_2 powder having an average grain diameter of 1.2 μm , a spherical SiO_2 powder having an average grain diameter of 0.7 μm , MnO powder having an average grain diameter of 0.8 μm , Co_3O_4 powder having an average grain diameter of 2 μm were prepared as oxide powders. Further, Co coarse powder of which grain diameter adjusted to fall within the range of 50 μm to 300 μm was prepared, and the weight ratio of the Co powder having an average grain diameter of 4 μm to the Co coarse powder was 7:3. The powders were weighed out in this composition ratio to give 2000 g.

Composition: 63Co-21Pt-3B-6 SiO_2 -2 TiO_2 -4MnO-1 Co_3O_4 (mol%)

[0038] The weighed powders, except for the Co coarse powder, were subsequently sealed in a 10-liter ball mill pot together with tungsten alloy balls as grinding media and rotated for 120 hours for mixing. Then, the Co coarse powder was further added to the ball mill pot, and mixed for 1 hour. The obtained mixed powder was filled in a carbon mold, and hot pressed under conditions of a vacuum atmosphere, a temperature of 1000°C, a holding time of 2 hours and a pressure of 30 MPa to obtain a sintered compact. This was cut with a lathe into a disk-shaped target having a diameter of 180 mm and a thickness of 5 mm.

[0039] The average leakage magnetic flux density of the target in Example 3 was 31%. Structural observation of a polished face of the target with a microscope showed that metal grains were scattered in a structure in which nonmagnetic material grains were dispersed in a metal phase. The number of metal grains having a sum of the maximum and the minimum diameter of 30 μm or more in 1 mm^2 field of view was 18 on average as evaluated in Example 1. Further, the ratio of nonmagnetic material grains having a difference between the

maximum and the minimum diameter of 0.7 μm or less as evaluated in Example 1 was 60%, and the average grain diameter was 1.16 μm .

[0040] The target was subsequently placed in DC magnetron sputtering device to perform sputtering. The sputtering conditions were a sputtering power of 1.2 kW, an Ar gas pressure of 1.5 Pa, and pre-sputtering was performed at 2 kW/hr as in Example 1. Then sputtering was performed on a silicon substrate having a diameter of 4 inches at a target deposition thickness of 1000 nm. The number of particles adhering on the substrate was counted with a particle counter, and the number of particles on the silicon substrate was 5.

(Example 4)

[0041] Fe powder having an average grain diameter of 4 μm , Pt powder having an average grain diameter of 3 μm , Fe-B powder having an average grain diameter of 7 μm were prepared as metal raw powders, and a spherical SiO_2 powder having an average grain diameter of 0.8 μm was prepared as an oxide powder. Further, Fe coarse powder adjusted to have the grain diameter in the range of 50 μm to 300 μm was prepared, and the weight ratio of the Fe powder having an average grain diameter of 4 μm to the Fe coarse powder was 8:2. These powders were weighed out in this composition ratio to give 2000 g.

Composition: 52Fe-25Pt-5B-18 SiO_2 (mol%)

[0042] The weighed powders, except for the Fe coarse powder, were subsequently sealed in a 10-liter ball mill pot together with tungsten alloy balls as grinding media and rotated for 120 hours for mixing. Then, the Fe coarse powder was further added to the ball mill pot, and mixed for 1 hour. The obtained mixed powder was filled in a carbon mold, and hot pressed under conditions of a vacuum atmosphere, a temperature of 1300°C, a holding time of 2 hours and a pressure of 30 MPa to obtain a sintered compact. This was cut with a lathe into a disk-shaped target having a diameter of 180 mm and a thickness of 5 mm.

[0043] The average leakage magnetic flux density of the target in Example 4 was 61%. Structural observation of a polished face of the target with a microscope showed that metal grains were scattered in a structure in which nonmagnetic material grains were dispersed in a metal phase. The number of metal grains having a sum of the maximum and the minimum diameter of 30 μm or more in 1 mm^2 field of view was 4 on average as evaluated in Example 1. Further, the ratio of nonmagnetic material grains having a difference between the maximum and the minimum diameter of 0.7 μm or less as evaluated in Example 1

was 65%, and the average grain diameter was 1.29 μm .

[0044] The target was subsequently placed in DC magnetron sputtering device to perform sputtering. The sputtering conditions were a sputtering power of 1.2 kW, an Ar gas pressure of 1.5 Pa, and pre-sputtering was performed at 2 kWhr as in Example 1. Then sputtering was performed on a silicon substrate having a diameter of 4 inches at a target deposition thickness of 1000 nm. The number of particles adhering on the substrate was counted with a particle counter, and the number of particles on the silicon substrate was 6.

(Comparative Example 1)

[0045] Co powder having an average grain diameter of 4 μm , Cr powder having an average grain diameter of 5 μm , Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and TiO_2 powder having an average grain diameter of 1.2 μm , a core-like SiO_2 powder having an average grain diameter of 0.7 μm , a Cr_2O_3 powder having an average grain diameter of 1 μm were prepared as oxide powders. Then, the powders were weighed out in this composition ratio to give 2000.

Composition: 69Co-18Pt-2Cr-5SiO₂-2TiO₂-4Cr₂O₃ (mol%)

[0046] Subsequently, the weighed powders were sealed in a 10-liter ball mill pot along with tungsten alloy balls as grinding media and rotated for 120 hours for mixing. The obtained mixed powder was filled in a carbon mold, and hot pressed under conditions of a vacuum atmosphere, a temperature of 1100°C, a holding time of 2 hours and a pressure of 30 MPa as in Example 1 to obtain a sintered compact. This was cut with a lathe into a disk-shaped target having a diameter of 180 mm and a thickness of 5 mm.

[0047] The average leakage magnetic flux density of the target in Comparative Example 1 was 18%. Structural observation of a polished face of the target with a microscope showed that the number of metal grains having a sum of the maximum and the minimum diameter of 30 μm or more in 1 mm² field of view was less than 1 on average as evaluated in Example 1. Further, the ratio of nonmagnetic material grains having a difference between the maximum and the minimum diameter of 0.7 μm or less as evaluated in Example 1 was 89%, and the average grain diameter was 0.71 μm .

[0048] The target was subsequently placed in DC magnetron sputtering device to perform sputtering. The sputtering conditions of a sputtering power of 1.2 kW and an Ar gas pressure of 1.5 Pa as in Example 1 were carried out, but a stable electric discharge was not obtained. Then, an electric discharge was

stabilized at a sputtering power of 1.7 kW and an Ar gas pressure of 2.8 Pa, and pre-sputtering was performed at 2 kWhr. Further sputtering was performed on a silicon substrate having a diameter of 4 inches at a target deposition thickness of 1000 nm. The number of particles adhering on the substrate was counted with a particle counter, and the number of particles on the silicon substrate was 9.

(Comparative Example 2)

[0049] Co powder having an average grain diameter of 4 μm , Cr powder having an average grain diameter of 5 μm , Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and TiO_2 powder having an average grain diameter of 1.2 μm , a core-like SiO_2 powder having an average grain diameter of 0.7 μm , Cr_2O_3 powder having an average grain diameter of 1 μm were prepared as oxide powders. Further, Co coarse powder adjusted to have the grain diameter in the range of 50 μm to 300 μm was prepared, and the weight ratio of the Co powder having an average grain diameter of 4 μm to the Co coarse powder was 7:3. The powders were weighed out in this composition ratio to give 2000 g.

Composition: 69Co-18Pt-2Cr-5SiO₂-2TiO₂-4Cr₂O₃ (mol%)

[0050] The weighed powders, except for the Co coarse powder, were subsequently sealed in a 10-littler ball mill pot together with tungsten alloy balls as grinding media and rotated for 70 hours for mixing. Then, the Co coarse powder was further added to the ball mill pot, and mixed for 1 hour. The obtained mixed powder was filled in a carbon mold, and hot pressed under conditions of a vacuum atmosphere, a temperature of 1100°C, a holding time of 2 hours and a pressure of 30 MPa to obtain a sintered compact. This was cut with a lathe into a disk-shaped target having a diameter of 180 mm and a thickness of 5 mm.

[0051] The average leakage magnetic flux density of the target in Comparative Example 2 was 29%. Structural observation of a polished face of the target with a microscope showed that the number of metal grains having a sum of the maximum and the minimum diameter of 30 μm or more in 1 mm² field of view was 36 on average as evaluated in Example 1. Further, the ratio of nonmagnetic material grains having a difference between the maximum and the minimum diameter of 0.7 μm or less as evaluated in Example 1 was 54%, and the average grain diameter was 1.87 μm .

[0052] The target was subsequently placed in DC magnetron sputtering device to perform sputtering. The sputtering conditions were a sputtering power of 1.2

kW, an Ar gas pressure of 1.5 Pa, and pre-sputtering was performed at 2 kWhr as in Example 1. Then sputtering was performed on a silicon substrate having a diameter of 4 inches at a target deposition thickness of 1000 nm. The number of particles adhering on the substrate was counted with a particle counter, and the number of particles on the silicon substrate was as many as 28.

5

[0053]

[Table 1]

	Composition of Target (mol%)	Average grain diameter of nonmagnetic material grain in sintered compact (μm)	Ratio of nonmagnetic material grain, of which difference of maximum and minimum diameter is $0.7\mu\text{m}$ or less (%)	Number of metal grains, of which sum of maximum and minimum diameter is $30\mu\text{m}$ or more	Abnormal discharge	Number of particle	PTF (%)
Example 1	69Co-18Pt-2Cr-5SiO ₂ -2TiO ₂ -4Cr ₂ O ₃	0.75	85	40	none	4	30
Example 2	61.2Co-22Pt-3Ru-0.8Ta-6SiO ₂ -2TiO ₂ -4CoO-1B ₂ O ₃	1.26	64	19	none	4	28
Example 3	63Co-21Pt-3B-6SiO ₂ -2TiO ₂ -4MnO-1Co ₃ O ₄	1.16	60	18	none	5	31
Example 4	Fe-25Pt-5B-18SiO ₂	1.29	65	4	none	6	61
Comparative Example 1	69Co-18Pt-2Cr-5SiO ₂ -2TiO ₂ -4Cr ₂ O ₃	0.71	89	0	occurred	9	18
Comparative Example 2	69Co-18Pt-2Cr-5SiO ₂ -2TiO ₂ -4Cr ₂ O ₃	1.87	54	36	none	28	29

[Industrial Applicability]

[0054] According to the present invention, the leakage magnetic field at the time of sputtering can be improved, and abnormal discharge due to a nonmagnetic material can be controlled by adjusting the structure of a sputtering target, in particular the shape of a nonmagnetic material grain and a metal grain. Therefore, when the target of the present invention is used, a self electric discharge can be obtained during sputtering with magnetron sputtering device. Further, it is useful as a ferromagnetic sputtering target used for depositing a magnetic thin film of a magnetic recording medium, in particular for depositing a hard disk drive recording layer because of the following excellent effects: an abnormal discharge due to a nonmagnetic material can be controlled; particle generation during sputtering caused by an abnormal discharge can be decreased; and a cost improving effect by improved yields can be obtained.

CLAIMS

1. A sintered sputtering target made from a material having nonmagnetic material grains dispersed in a magnetic material containing Co or Fe, comprising:

5 a structure observed on a polished surface of the target comprises nonmagnetic material grains having an average grain diameter of 1.8 μm or less, a metal phase containing Co or Fe in which nonmagnetic material grains are dispersed and metal grains containing Co or Fe;

10 nonmagnetic material grains having a difference of 0.7 μm or less between a maximum diameter and a minimum diameter is 60% or more of nonmagnetic material grains in a structure observed on a polished surface of the target, defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of the nonmagnetic material grain and a minimum diameter as the smallest value of distances between two parallel lines
15 when the two lines sandwich the grain; and

one or more metal grains having a sum of the maximum diameter and the minimum diameter of 30 μm or more are present on average in 1 mm^2 field of view, defining a maximum diameter as the greatest value of distances between any two points on the outer periphery of the metal grain, and a minimum
20 diameter as the smallest value of distances between two parallel lines when the two lines sandwich the metal grain.

2. The sputtering target according to claim 1, wherein the nonmagnetic material grain is one or more oxides selected from B_2O_3 , CoO , Co_3O_4 , MnO , Mn_3O_4 , SiO_2 , SnO_2 , TiO_2 , Ti_2O_3 , Cr_2O_3 , Ta_2O_5 , WO_2 , WO_3 , ZrO_2 in an amount of
25 0.5 or more and 20 mol% or less.

3. The sputtering target according to claim 1 or 2, wherein in the case of Co based alloy used for the sputtering target, the sputtering target further comprises Cr at 0 mol% to 15 mol%, Pt at 5 mol% or more and 30 mol% or less, the remainder being Co and unavoidable impurities except for the nonmagnetic material.
30

4. The sputtering target according to claim 3, further comprising one or more elements selected from Mg, Al, Si, Mn, Nb, Mo, Ru, Pd, Ta, W, B in an amount of 0.5 mol% or more and 12 mol% or less.