MAGNETIC MATERIAL SPUTTERING TARGET AND METHOD FOR MANUFACTURING SAME

Technical Field

[0001] The present invention relates to a magnetic sputtering target suitable for depositing a magnetic thin film used for a recording layer of a magnetic recording medium, for example, a granular film of a magnetic recording medium of a hard disk adopting a vertical magnetic-recording system; and in particular relates to a magnetic sputtering target and method for manufacturing the sputtering target, capable of suppressing the generation of abnormal discharge and preventing the generation of particles during sputtering.

Background Art

[0002] With a magnetic recording medium such as a hard disk, a thinned magnetic body material formed on a substrate made from glass or the like is used as the magnetic recording layer; and the deposition of the magnetic recording layer is carried out by a magnetron sputtering method using a DC (dc) power source, of which method is widely adopted. In the magnetron sputtering method, a magnet is arranged on the back surface of a target, and magnetic flux is leaked to the target surface, the charged particles in the discharge plasma are restricted by the Lorentz force to the magnetic flux, a high-density plasma can be concentrated in the vicinity of the target surface, thus the film formation speed can be increased.

[0003] In the field of magnetic recording represented by hard disk drives, as a material of a magnetic thin film serving as a magnetic recording layer for recording, Co, Fe, or Ni based on a ferromagnetic metal is used. For example, a Co-Cr-based or Co-Cr-Pt-based ferromagnetic alloy, which has Co as its main component, has been conventionally used as a recording layer of a hard disk adopting the in-plane magnetic recording system in which the magnetization direction of the magnetic body is set to a direction that is parallel to the recording surface.

[0004] Meanwhile, a vertical magnetic-recording system which achieves high densification of the magnetic recording amount per recording area by setting the magnetization direction of the magnetic body to a direction that is vertical to the recording surface has been put into practical application, and has become mainstream in recent years. As the magnetic recording layer of a hard disk

adopting the vertical magnetic-recording system, a complex material configured from a Co-Cr-Pt-based ferromagnetic alloy, which has Co as its main component, and a nonmagnetic inorganic substance, is frequently used. Further, a magnetic thin film of a magnetic recording medium such as a hard disk has high productivity, a magnetic material sputtering target containing the above-mentioned material as a component is often prepared by sputtering.

[0005] As the method of preparing this kind of magnetic sputtering target, the melting method or the powder metallurgy method can be considered. Which method should be used for preparing a sputtering target cannot be decided unconditionally because it will depend on the required sputtering characteristics and the thin film performance; however, the powder metallurgy method is generally used to prepare a sputtering target used for forming a recording layer of a hard disk of the vertical magnetic-recording system which is becoming mainstream in recent years. This is because, with a sputtering target for forming a recording layer of the vertical magnetic-recording system, it is necessary to uniformly disperse inorganic grains in an alloy base, but it is difficult for the melting method to realize this kind of structure.

[0006] Approaches have been taken for improving the production of a magnetic sputtering target based on powder metallurgy from several perspectives. For example, Patent Documents 1 and 2 disclose a sintered sputtering target in which oxide grains are dispersed in an alloy based on powder metallurgy, and describe that, by causing an alloy having a specific elemental composition to exist as coarsened grains in the alloy base, it is possible to reduce the magnetic permeability of the overall target, increase the path through flux (PTF) that passes through to the sputter surface of the magnetic target, increase the plasma density near the sputter surface, and thereby improve the deposition rate.

[0007] Patent Documents 3 and 4, from a different perspective, disclose a sintered sputtering target obtained by dispersing oxide grains in an alloy based on powder metallurgy and sintering the resulting product, and further disclose a technology of obtaining a fine and uniform texture by controlling the shape and dispersion morphology of the oxides dispersed in the target. In these targets, because the oxides which are dispersion elements are insulators, they may become the cause of abnormal discharge depending on the shape and dispersion morphology thereof; thus abnormal discharge is suppressed and generation of particles is prevented by causing the texture of the target to be fine and uniform.

[0008] Nevertheless, even with these conventional technologies, there is room

for further improvement of the existence form and dispersion morphology of oxides in the target, and a sputtering target capable of more effectively suppressing abnormal discharge and preventing the generation of particles was desired. In particular, ever since the vertical recording system became mainstream, the floating quantity of the magnetic head in a magnetic recording device such as a hard disk drive is becoming smaller each year pursuant to the improvement in recording density, and requirements of the size and number of particles that are tolerated in a magnetic recording medium are becoming even stricter.

Prior Art Documents

Patent Documents

[0009]

[Patent Document 1] Japanese Patent No. 5375707

[Patent Document 2] International Publication No. 2014/125897

[Patent Document 3] International Publication No. 2013/125469

[Patent Document 4] Japanese Patent No. 4975647

SUMMARY OF THE INVENTION

Problems to be Solved by the Invention

[0010] The pesent invention relates to providing a magnetic sputtering target in which nonmagnetic material grains are dispersed, and a method for producing the same, capable of effectively reducing the generation of abnormal discharge and particles caused by oxides, particularly coarsely grown oxides in the sputtering target.

Means for Solving the Problems

[0011] The present inventors, as a result of intense study to achieve the foregoing object, discovered that when oxides having a low-melting-point are included in the sputtering target, there is a problem in that such oxides will melt, become aggregated, or react with other oxides during the sintering step, and grow to become aggregates with an extremely large grain size, and it is possible to suppress the generation of such aggregates by performing heat treatment to the oxides in advance prior to the sintering step, and it is thereby possible to reduce the amount of particles that are generated due to oxides during sputtering.

[0012] Based on the foregoing discovery, the invention is explained as follows:

1) A magnetic sputtering target including oxides having a melting point of 500°C or lower, which includes an average number density of oxides having a grain size of 10 μm or more on a sputter surface of the sputtering target is $5/mm^2$ or less.

2) The magnetic sputtering target according to 1) above, wherein the sputtering target includes oxides has one or more types selected from Cr, Ta, Ti, Si, Zr, Al, Nb, and Co as its constituent.

3) The magnetic sputtering target according to 1) or 2) above, wherein a total content of oxides in the sputtering target is 5 vol% or more and 50 vol% or less.

4) The magnetic sputtering target according to any one of 1) to 3) above, wherein Co is 55 mol% or more and 95 mol% or less, Cr is 40 mol% or less, and Pt is 45 mol% or less in the sputtering target.

5) The magnetic sputtering target according to 4) above, wherein the sputtering target includes 10 mol% or less of one or more types selected from B, N, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Si, and Al.

6) A production method of the magnetic sputtering target according to any one of 1) to 5) above, wherein an oxide powder containing oxides having a melting point of 500°C or lower is heat treated at a temperature that is equal to or higher than a sintering temperature of a target, and the heat-treated powder is used as a sintering material.

7) A production method of the magnetic sputtering target according to any one of 1) to 5) above, wherein an oxide powder excluding oxides having a melting point of 500°C or lower is heat treated at a temperature that is equal to or higher than a sintering temperature of a target, and the heat-treated powder is used as a sintering material.

8) The production method of a magnetic sputtering target according to 6) or 7) above including a step of performing heat treatment in an atmosphere at 800°C or higher and 1900°C or lower.

9) The production method of a magnetic sputtering target according to 6) above including a step of adjusting a grain size of the heat-treated oxide powder to be an average grain size of 5 μm or less.

10) The production method of a magnetic sputtering target according to any one of 6) to 9) above including a step of performing hot press sintering at a holding temperature of 500°C to 1400°C.

Effect of the Invention

[0013] The magnetic sputtering target of the embodiments of the present invention with nonmagnetic material grains dispersed therein can contribute to the suppression of abnormal discharge and the reduction of particle generation caused by coarse oxides during sputtering, and can considerably improve the characteristics compared to conventional sputtering targets. The present invention consequently yields a superior effect of cost improvement brought by further imorvement of yield.

BRIEF DESCRIPTION OF THE DRAWINGS

[0014] Fig. 1 is a schematic diagram for explaining the grain size of the oxide grains according to an embodiment of the present invention.

Fig. 2 is a diagram showing the positions where the texture of the sputtering target according to an embodiment of the present invention was observed.

Fig. 3 is an image of the texture of the sputtering target (sputter surface side) of Example 1 observed with a laser microscope.

Fig. 4 is an image of the texture of the sputtering target (sputter surface side) of Comparative Example 1 observed with a laser microscope.

Fig. 5 is an EPMA elemental mapping image of the oxides in the sputtering target of Comparative Example 1.

DESCRIPTION OF EMBODIMENTS

[0015] When oxides having a melting point of 500°C or lower (these may be hereinafter referred to as “low-melting-point oxides”) are used as a raw material of a nonmagnetic material, these low-melting-point oxides would sometimes melt during sintering, or form aggregates due to reaction with themselves or other oxides. When oxides become aggregated, and coarsened oxides exist in the sintered body (sputtering target), abnormal discharge may occur with such oxides as the origin during sputtering, or particles may become generated as a result of coarsened oxides falling off. The said abnormal discharge or particles caused deterioration in the film quality and further reduces the yield of the product.

[0016] An embodiment of the present invention is characterized in that by performing heat treatment to the oxides in advance, it is possible to suppress the formation of aggregates caused by the melting of oxides during sintering, and reduce

the existence ratio of coarse oxides in the sputtering target. In other words, the magnetic sputtering target of the embodiment of the present invention is characterized in that, when it includes oxides having a melting point of 500°C or lower as nonmagnetic material grains, the average number density of oxides having a grain size of 10 μm or more existing in the sputtering target is caused to be 5/mm^2 or less.

[0017] The shape of oxides (schematic plan view) existing in the sputtering target is shown in Fig. 1. As shown in Fig. 1, because the planar shape of oxides is not necessarily a perfect circle or oval shape, in the embodiment of the present invention, the diameter of the maximum inscribed circle drawn in a planar shape of oxides is defined as the grain size. Fig. 2 shows a schematic diagram showing the positions where the texture of the sputtering target was observed. As shown in Fig. 2, the texture was observed at a total of 10 positions including the center and points that are 1/2 of the radius (r) of the target, and the average value of the number density of oxides in each observed texture was used as the average number density. Here, in order to accurately comprehend the shape of oxides, observation was performed with a field of view of 1075 μm × 1433 μm.

[0018] When oxides having a melting point of 500°C or lower are used, the aggregation phenomenon becomes notable during sintering. As an oxide having a melting point of 500°C or lower, for instance, boron oxide (B_2O_3) may be considered. Because B_2O_3 is a material often used as a nonmagnetic material of a magnetic sputtering target, an embodiment of the present invention makes reference to B_2O_3. However, because the same phenomenon will occur with any oxide having a melting point of 500°C or lower, the embodiment of the present invention can be applied even when using low-melting-point oxides other than B_2O_3 as the sintering material.

[0019] When oxides having a melting point of 500°C or lower (low-melting-point oxides) are included in the sintering material, they will melt during sintering and remain as coarse oxide aggregates in the sintered body. As a means for suppressing the generation of these aggregates, the followings may be adapted, 1) heat treating the low-melting-point oxides together with other oxides and synthesizing compounds (complex oxides) having different melting points, or 2) heat treating oxides other than the low-melting-point oxides in advance to make it difficult for a reaction to occur with (to lower the reactivity of) low-melting-point oxides during sintering.

[0020] As oxides other than the low-melting-point oxides, considered may be oxides having one or more types selected from Cr, Ta, Ti, Si, Zr, Al, Nb, and Co as

its constituent. These oxides exist in the sputtering target as single element oxides, or complex oxides of the elements or as complex oxides synthesized with the low-melting-point oxides. The total content of oxides including the low-melting-point oxides in the sputtering target is preferably 5 vol% or more and 50 vol% or less. Favorable magnetic properties can be obtained by causing the total volume ratio of oxides to be 5 vol% or more. Oxides can be uniformly and finely dispersed, by causing the total volume ratio of oxides to be 50 vol% or less. More preferably, the total volume ratio of oxides is 20 vol% or more and 40 vol% or less.

[0021] The magnetic sputtering target according to the embodiment of the present invention contains Co at 55 mol% or more and 95 mol% or less in the sputtering target, and may additionally contain, as arbitrary components, Pt at 45 mol% or less and Cr at 40 mol% or less, and Pt and Cr may also be 0 mol%. The composition is mainly determined based on the magnetic properties required in the magnetic recording layer. In order to more strictly control the magnetic properties, preferably, the content of Co is set at 60 mol% or more and 85 mol% or less, the content of Pt at 25 mol% or less, and the content of Cr at 20 mol% or less. In order to improve the magnetic properties, it would be effective to include 10 mol% or less of one or more types selected from B, N, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Si, and Al.

[0022] The magnetic sputtering target of the present invention can be prepared, for instance, based on the powder sintering method described below.

Prepare Co powder, Pt powder, and Cr powder as the magnetic materials, and additionally add powders of B, Ti, V and the like as additives. In addition to these powders, an alloy powder may be used as well as a single-element powder. Preferably, powders having a grain size in a range of 1 to 10 μm are used. When the grain size of the powder is 1 to 10 μm, more uniform mixing is possible, thereby can prevent segregation and crystal coarsening. When the grain size of the metal powder is greater than 10 μm, there are cases where the nonmagnetic materials are not dispersed uniformly; on the contrary, when the grain size of the metal powder is smaller than 1 μm, there is a problem in that the composition of the target deviates from the intended composition due to the influence of the oxidation of the metal powder. Incidentally, this grain size range is merely a preferred range, and it should be understood that the deviation from the foregoing grain size range is not a condition to deny the present invention.

[0023] A nonmagnetic material including oxides having a melting point of 500°C or lower, and a powder of oxide having Cr, Ta, Ti, Si or the like, as its constituent.

Preferably oxides having a grain size in a range of 1 to 5 μm are used. When the grain size of oxides is equal to or smaller than the grain size of metal powders, it is easy to pulverize the oxides, and the oxides can be uniformly dispersed, since upon mixing the oxides with the foregoing metal powders, the nonmagnetic material powders do not become aggregated easily. This grain size range is merely a preferred range, and it should be understood that the deviation from the foregoing grain size range is not a condition to deny the present invention.

[0024] The advance heat treatment of oxides, which is a key point of the aspects of the invention, is now explained. With the foregoing issue that oxides having a melting point of 500°C or lower will melt during the sintering of the target and tend to generate aggregates. Thus, according to embodiments of the present invention, by performing advance heat treatment to the oxides at a temperature higher than the sintering temperature of the target, specifically, 1) the low-melting-point oxide is synthesized into a composite oxide having a higher melting point; 2) the reactivity of the oxide other than the low-melting-point oxide is lowered.

[0025] In order to achieve 1) above, mixed powders of oxides which have a melting point of 500°C or lower, i.e. low-melting-point oxides and other oxides, i.e. oxides having a melting point exceeding 500°C are heat treated at a temperature higher than the sintering temperature of the target. Because of the heat treatment, the low-melting-point oxides become synthesized with other oxides to form complex oxides having a higher melting point; and the aggregation associated with the melting of oxides during the sintering of the target is suppressed.

In order to achieve 2) above, only oxides (oxides having a melting point exceeding 500°C) other than oxides having a melting point of 500°C or lower, i.e. low-melting-point oxides are heat treated at a temperature higher than the sintering temperature of the target. Because of the heat treatment, reaction with the low-melting-point oxides during the sintering of targets is suppressed and the coarsening of the oxides is also suppressed.

[0026] The heat treatment of an oxide powder is performed at a temperature higher than the sintering temperature of the target; and preferably, in the atmosphere at 800°C or higher and 1900°C or lower. When the heat treatment temperature is less than 800°C, it is possible that the effect of heat treatment of the oxide powder may be insufficient; on the other hand, when the heat treatment temperature exceeds 1900°C, it is undesirable because of the increase of the energy cost. Preferably, the heat treatment time of the oxide powder is 2 hours or longer. After the heat

treatment of the oxide powder, the oxide powder are preferably pulverized using a mortar or the like to adjust the grain size so that the average grain size will be 5 μm or less. When the average grain size is within a range of 5 μm or less, the nonmagnetic material powders will not become aggregated easily when mixed with the foregoing metal powders, but they can be uniformly dispersed.

[0027] Next, the foregoing raw material powders and the heat-treated the oxide powder are weighed to attain the intended composition, and pulverized and mixed using a publicly known method such as a ball mill. Subsequently, the obtained mixed powder is molded and sintered via the hot press method in a vacuum atmosphere or an inert gas atmosphere. In addition to the hot press method, various pressure sintering methods such as the plasma discharge sintering method may also be used. In particular, the hot isostatic sintering method is effective in improving the density of the sintered body. The holding temperature during the sintering of the target is preferably set, while this also depends on the component composition of the target, to fall within a temperature range of 500°C to 1400°C. The sputtering target according to the embodiments of the present invention can be produced by processing the thus obtained sintered body into the intended shape using a lathe.

[0028] The evaluation method of the embodiments of the present invention, including the Examples and Comparative Examples described below, is as follows.

(Texture observation of target and number density of oxide grains)

Evaluation of the texture of the target surface was performed using an enlarged image from a laser microscope. The surface texture of the target that underwent pretreatment such as polishing and washing was observed using a laser microscope at a total of 10 positions including the center (1 point) and points (9 points) that are 1/2 of the radius of the target as shown in Fig. 2, and each observation image was captured. The observation magnification was set to a field of view area of 1075 μm × 1433 μm so as to accurately evaluate the shape of oxides. Next, the extracted texture images of the 10 positions were converted into a binary image. The threshold for binarization was set within the range of the contrast difference between the matrix having a metal component as its main component and the boundries of the oxide grains. Normally, while it is possible to clearly differentiate the boundary of the two based on the contrast difference between the matrix and the oxides, the sharpness of separation can be improved by concurrently

performing processes based on the discriminant analysis method, differential histogram method or the like. Subsequently, with respect to the binarized images of the 10 positions, the number of oxide grains having a grain size of 10 μm or more was counted, the number density per unit area obtained by dividing the number of oxide grains by the observation field of view area was calculated, and the average value (number density) of the 10 positions was obtained.

(Volume ratio of oxides in target)

[0029] In the embodiment of the present invention, the volume ratio of oxides in the sputtering target was evaluated as a value corresponding to the area ratio of oxides in the overall observation field of view (area ratio [%] = oxide area [μm^2] obtained based on binarization analysis / field of view area [μm^2] × 100) in the foregoing observation images from a laser microscope. The area ratio of oxides in the overall observation field of view is actually the ratio of the area indicating the oxides in a two-dimensional plane, and is not the ratio of the volume in a three-dimensional space, and the two-dimensional area ratio can be deemed the volume ratio in a three-dimensional space based on the premise that particles are dispersed isotropically in all orientations. The volume ratio (vol. %) of oxides evaluated from the observation images is not considerably different from the volume ratio of oxides evaluated from the weight and density of the raw material.

Examples

[0030] The embodiments of the present invention are now specifically explained based on the Examples and Comparative Examples. The descriptions in the following Examples are specific examples for facilitating the understanding of the technical subject matter of the present invention, and the technical scope of the present invention is not limited by these specific examples.

Example 1 and Comparative Example 1

[0031] Prepared were Co powder having an average grain size of 3 μm, Pt powder having an average grain size of 3 μm, and Cr powder having an average grain size of 3 μm as the raw material powders of metal components; and B_2O_3 powder having an average grain size of 1 μm, TiO_2 powder having an average grain size of 1 μm, SiO_2 powder having an average grain size of 1 μm, Cr_2O_3 powder having an average grain size of 1 μm, and CoO powder having an average grain size

of 1 μm as the raw material powders of oxide components. These powders were weighed to attain the following mol ratio composition. The composition is as follows.

Composition: 70Co - 4Cr - 10Pt - 4B_2O_3 - 2TiO_2 - 2SiO_2 - 2Cr_2O_3 - 6CoO mol%

[0032] In Example 1, the two types of the oxide powder of TiO_2 powder and SiO_2 powder, which are raw material powders of oxide components, were mixed, and the obtained mixed powder was subject to heat treatment. Heat treatment was performed in an ordinary pressure atmosphere at 1050°C for 5 hours. The heat-treated oxide powder was once cooled to room temperature via furnace cooling, and provided to the subsequent mixing step. Note that heat treatment was not performed in Comparative Example 1.

[0033] Next, the oxide powder that underwent heat treatment (only Example 1), the oxide powder without heat treatment, and the raw material powders of metal components were mixed and pulverized for 10 minutes with a planetary motion type mixer with a ball capacity of approximately 7 liters, placed in a 10-liter ball mill pot together with TiO_2 balls as the pulverizing medium, and rotated and mixed for 20 hours. Next, the obtained mixed powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions; temperature of 850°C, holding time of 2 hours, and pressure of 30 MPa, to obtain a sintered body. Further, the sintered body was cut to obtain a disc-shaped sputtering target having a diameter of 165.1 mm and a thickness of 5 mm.

[0034] The surface of the obtained sputtering target was polished, then the texture of the sputtering target was observed with a laser microscope. Fig. 3 (Example 1) and Fig. 4 (Comparative Example 1) respectively show the obtained texture image. Moreover, the average number of grains of oxides having a grain size of 10 μm or more in 10 fields of view and existing in the texture images of each field of view area of 1075 μm × 1433 μm was 2.9 and the average number density was 1.88 /mm^2 in Example 1, which satisfied the range of the embodiment of the present invention. Meanwhile, the average number of grains was 12.5, and the average number density was 8.11 /mm^2 in Comparative Example 1; both values deviated from the range of the embodiment of the present invention.

Here, the oxides in the sputtering target of Comparative Example 1 were shown using the elemental mapping obtained from an electron probe microanalyzer (EPMA). As shown in Fig. 5, it has been confirmed that the oxides are complex oxides configured from Co-B-O, Si-B-O. This is considered to be a result of B_2O_3

melting and forming aggregates during sintering.

[0035] Next, the sputtering target was mounted on a DC magnetron sputtering device and sputtered, and particle evaluation was performed. The sputtering conditions were input power of 1 kW, sputtering time of 20 seconds, and Ar atmospheric pressure of 1.7 Pa. Subsequently, the number of particles having a diameter of 0.07 μm or more that adhered to the substrate was measured using a particle counter. Consequently, the number of particles in Example 1 was 51 and 129 in Comparative Example 1; thus a significant difference was confirmed.

Example 2 and Comparative Example 2

[0036] Prepared were Co powder having an average grain size of 3 μm and Pt powder having an average grain size of 3 μm as the raw material powders of metal components; and B_2O_3 powder having an average grain size of 1 μm, TiO_2 powder having an average grain size of 1 μm, and SiO_2 powder having an average grain size of 1 μm as the raw material powders of oxide components. These powders were weighed to attain the following mol ratio composition. The composition is as follows.

Composition: 65Co - 20Pt - $5B_2O_3$ - $5TiO_2$ - $5SiO_2$ mol%

In Example 2, the two types of the oxide powder of TiO_2 powder and SiO_2 powder, which are raw material powders of oxide components, were mixed, and the obtained mixed powder was subject to heat treatment. The conditions of heat treatment were the same as Example 1. The heat-treated oxide powder was once cooled to room temperature via furnace cooling, and provided to the subsequent mixing step. Note that in Comparative Example 2, heat treatment was not performed.

[0037] Next, the oxide powder that underwent heat treatment (only Example 2), the oxide powder without heat treatment, and the raw material powders of metal components were mixed and pulverized for 10 minutes with a planetary motion type mixer having a ball capacity of approximately 7 liters, placed in a 10-liter ball mill pot together with TiO_2 balls as the pulverizing medium, and rotated and mixed for 20 hours. Next, the obtained mixed powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions; temperature of 850°C, holding time of 2 hours, and pressure of 30 MPa, to obtain a sintered body. Further, the sintered body was cut to obtain a disc-shaped sputtering target having a diameter of 165.1 mm and a thickness of 5 mm.

[0038] As a result of observing the surface texture of the obtained sputtering

target in the same manner as Example 1, the average number of grains of oxides having a grain size of 10 μm or more in 10 fields of view and existing in the texture images of each field of view area of 1075 μm × 1433 μm was 7.0, and the average number density was 4.54/mm^2 in Example 2; which satisfied the range of the embodiment of the present invention. Meanwhile, the average number of grains was 10.0, and the average number density was 6.49/mm^2 in Comparative Example 2; and both values deviated from the range of the embodiment of the present invention.

As a result of evaluating this target based on the same sputtering test as Example 1, the number of particles having a diameter of 0.07 μm or more observed on the silicon substrate was 76 in Example 2 and 88 in Comparative Example 2; thus a significant difference was confirmed.

Example 3 and Comparative Example 3

[0039] Prepared were Co powder having an average grain size of 3 μm and Cr powder having an average grain size of 3 μm as the raw material powders of metal components; and B_2O_3 powder having an average grain size of 1 μm, TiO_2 powder having an average grain size of 1 μm, and SiO_2 powder having an average grain size of 1 μm as the raw material powders of oxide components. These powders were weighed to attain the following mol ratio composition. The composition is as follows.

Composition: 65Co - 20Cr - 5B_2O_3 - 5TiO_2 - 5SiO_2 mol%

In Example 3, the three types of the oxide powder of B_2O_3 powder, TiO_2 powder and SiO_2 powder, which are raw material powders of oxide components, were mixed, and the obtained mixed powder was subject to heat treatment. The conditions of heat treatment were the same as Example 1. The heat-treated oxide powder was once cooled to room temperature via furnace cooling, and provided to the subsequent mixing step. Note that heat treatment was not performed in Comparative Example 3.

[0040] Next, the oxide powder that underwent heat treatment (only Example 3), the oxide powder without heat treatment, and the raw material powders of metal components were mixed and pulverized with a planetary motion type mixer having a ball capacity of approximately 7 liters, placed in a 10-liter ball mill pot together with TiO_2 balls as the pulverizing medium, and rotated and mixed for 20 hours. Next, the obtained mixed powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the conditions of temperature of 850°C, holding time of 2 hours, and pressure of 30 MPa, to obtain a sintered body. Further, the sintered body was

cut to obtain a disc-shaped sputtering target having a diameter of 165.1 mm and a thickness of 5 mm.

[0041] As a result of observing the surface texture of the obtained sputtering target in the same manner as Example 1, the average number of grains of oxides having a grain size of 10 μm or more in 10 fields of view and existing in the texture images of each field of view area of 1075 μm × 1433 μm was 3.5, and the average number density was 2.27/mm^2 in Example 3; which satisfied the range of the embodiment of the present invention. Meanwhile, the average number of grains was 11.2, and the average number density was 7.27/mm^2 in Comparative Example 3; and both values deviated from the range of the embodiment of the present invention.

As a result of evaluating this target based on the same sputtering test as Example 1, the number of particles having a diameter of 0.07 μm or more observed on the silicon substrate was 70 in Example 3 and 118 in Comparative Example 3; thus a significant difference was confirmed.

Example 4 and Comparative Example 4

[0042] Prepared were Co powder having an average grain size of 3 μm and Cr powder having an average grain size of 3 μm as the raw material powders of metal components; and B_2O_3 powder having an average grain size of 1 μm, and TiO_2 powder having an average grain size of 1 μm as the raw material powders of oxide components. These powders were weighed to attain the following mol ratio composition. The composition is as follows.

Composition: 65Co - 20Cr - 5B_2O_3 - 10TiO_2 mol%

In Example 4, the two types of the oxide powder of B_2O_3 powder and TiO_2 powder, which are raw material powders of oxide components, were mixed, and the obtained mixed powder was subject to heat treatment. Heat treatment was performed in an ordinary pressure atmosphere at 950°C for 5 hours. The heat-treated oxide powder was once cooled to room temperature via furnace cooling, and provided to the subsequent mixing step. Note that heat treatment was not performed in Comparative Example 4.

[0043] Next, the oxide powder that underwent heat treatment (only Example 4), the oxide powder without heat treatment, and the raw material powders of metal components were mixed and pulverized for 10 minutes with a planetary motion type mixer with a ball capacity of approximately 7 liters, placed in a 10-liter ball mill pot

together with TiO_2 balls as the pulverizing medium, and rotated and mixed for 20 hours. Next, the obtained mixed powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions; temperature of 850°C, holding time of 2 hours, and pressure of 30 MPa, to obtain a sintered body. Further, the sintered body was cut to obtain a disc-shaped sputtering target having a diameter of 165.1 mm and a thickness of 5 mm.

[0044] As a result of observing the surface texture of the obtained sputtering target in the same manner as Example 1, the average number of grains of oxides having a grain size of 10 μm or more in 10 fields of view and existing in the texture images of each field of view area of 1075 μm × 1433 μm was 7.2, and the average number density was 4.67/mm^2 in Example 4; which satisfied the range of the embodiment of the present invention. Meanwhile, the average number of grains was 15.5, and the average number density was 10.06/mm^2 in Comparative Example 4; and both values deviated from the range of the embodiment of the present invention.

As a result of evaluating this target based on the same sputtering test as Example 1, the number of particles having a grain size of 0.07 μm or more observed on the silicon substrate was 98 in Example 4 and 217 in Comparative Example 4; thus a significant difference was confirmed.

Example 5 and Comparative Example 5

[0045] Prepared were Co powder having an average grain size of 3 μm and Cr powder having an average grain size of 3 μm as the raw material powders of metal components; and B_2O_3 powder having an average grain size of 1 μm, and SiO_2 powder having an average grain size of 1 μm as the raw material powders of oxide components. These powders were weighed to attain the following mol ratio composition. The composition is as follows.

Composition: 65Co - 20Cr - 5B_2O_3 - 10SiO_2 mol%

In Example 5, the two types of the oxide powder of B_2O_3 powder and SiO_2 powder, which are raw material powders of oxide components, were mixed, and the obtained mixed powder was subject to heat treatment. Heat treatment was performed in an ordinary pressure atmosphere at 850°C for 5 hours. The heat-treated oxide powder was once cooled to room temperature via furnace cooling, and provided to the subsequent mixing step. Note that heat treatment was not

performed in Comparative Example 5.

[0046] Next, the oxide powder that underwent heat treatment (only Example 5), the oxide powder without heat treatment, and the raw material powders of metal components were mixed and pulverized for 10 minutes with a planetary motion type mixer with a ball capacity of approximately 7 liters, placed a 10-liter ball mill pot together with TiO_2 balls as the pulverizing medium, and rotated and mixed for 20 hours. Next, the obtained mixed powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions; temperature of 850°C, holding time of 2 hours, and pressure of 30 MPa, to obtain a sintered body. Further, the sintered body was cut to obtain a disc-shaped sputtering target having a diameter of 165.1 mm and a thickness of 5 mm.

[0047] As a result of observing the surface texture of the obtained sputtering target in the same manner as Example 1, the average number of grains of oxides having a grain size of 10 μm or more in 10 fields of view and existing in the texture images of each field of view area of 1075 μm × 1433 μm was 5.1, and the average number density was 3.31/mm^2 in Example 5; which satisfied the range of the embodiment of the present invention. Meanwhile, the average number of grains was 7.9, and the average number density was 5.13/mm^2 in Comparative Example 5; and both values deviated from the range of the embodiment of the present invention.

As a result of evaluating this target based on the same sputtering test as Example 1, the number of particles having a diameter of 0.07 μm or more observed on the silicon substrate was 66 in Example 5 and 77 in Comparative Example 5; thus a significant difference was confirmed.

Example 6 and Comparative Example 6

[0048] Prepared were Co powder having an average grain size of 3 μm and Cr powder having an average grain size of 3 μm as the raw material powders of metal components; and B_2O_3 powder having an average grain size of 1 μm, and Cr_2O_3 powder having an average grain size of 1 μm as the raw material powders of oxide components. These powders were weighed to attain the following mol ratio composition. The composition is as follows.

Composition: 65Co - 20Cr - 5B_2O_3 - 10Cr_2O_3 mol%

In Example 6, the two types of the oxide powder of B_2O_3 powder and Cr_2O_3 powder, which are raw material powders of oxide components, were mixed,

and the obtained mixed powder was subject to heat treatment. Heat treatment was performed in an ordinary pressure atmosphere at 850°C for 5 hours. The heat-treated oxide powder was once cooled to room temperature via furnace cooling, and provided to the subsequent mixing step. Note that heat treatment was not performed in Comparative Example 6.

[0049] Next, the oxide powder that underwent heat treatment (only Example 6), the oxide powder without heat treatment, and the raw material powders of metal components were mixed and pulverized for 10 minutes with a planetary motion type mixer with a ball capacity of approximately 7 liters, placed a 10-liter ball mill pot together with TiO_2 balls as the pulverizing medium, and rotated and mixed for 20 hours. Next, the obtained mixed powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions; temperature of 850°C, holding time of 2 hours, and pressure of 30 MPa, to obtain a sintered body. Further, the sintered body was cut to obtain a disc-shaped sputtering target having a diameter of 165.1 mm and a thickness of 5 mm.

[0050] As a result of observing the surface texture of the obtained sputtering target in the same manner as Example 1, the average number of grains of oxides having a grain size of 10 μm or more in 10 fields of view and existing in the texture image of each field of view area of 1075 μm × 1433 μm was 7.1, and the average number density was 4.61/mm^2 in Example 6; which satisfied the range of the embodiment of the present invention. Meanwhile, the average number of grains was 14.3, and the average number density was 9.28/mm^2 in Comparative Example 6; and both values deviated from the range of the embodiment of the present invention.

As a result of evaluating this target based on the same sputtering test as Example 1, the number of particles having a diameter of 0.07 μm or more observed on the silicon substrate was 102 in Example 6 and 182 in Comparative Example 6; thus a significant difference was confirmed.

Example 7 and Comparative Example 7

[0051] Prepared were Co powder having an average grain size of 3 μm and Cr powder having an average grain size of 3 μm as the raw material powders of metal components; and B_2O_3 powder having an average grain size of 1 μm; and Ta_2O_5 powder having an average grain size of 1 μm as the raw material powders of oxide components. These powders were weighed to attain the following mol ratio composition. The composition is as follows.

Composition: 65Co - 20Cr - $5B_2O_3$ – $10Ta_2O_5$ mol%

In Example 7, the two types of the oxide powder of B_2O_3 powder and Ta_2O_5 powder, which are raw material powders of oxide components, were mixed, and the obtained mixed powder was subject to heat treatment. Heat treatment was performed in an ordinary pressure atmosphere at 1050°C for 5 hours. The heat-treated oxide powder was once cooled to room temperature via furnace cooling, and provided to the subsequent mixing step. Meanwhile, heat treatment was not performed in Comparative Example 7.

[0052] Next, the oxide powder that underwent heat treatment (only Example 7), the oxide powder without heat treatment, and the raw material powders of metal components were mixed and pulverized for 10 minutes with a planetary motion type mixer with a ball capacity of approximately 7 liters, placed in a 10-liter ball mill pot together with TiO_2 balls as the pulverizing medium, and rotated and mixed for 20 hours. Next, the obtained mixed powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions; temperature of 850°C, holding time of 2 hours, and pressure of 30 MPa, to obtain a sintered body. Further, the sintered body was cut to obtain a disc-shaped sputtering target having a diameter of 165.1 mm and a thickness of 5 mm.

[0053] As a result of observing the surface texture of the obtained sputtering target in the same manner as Example 1, the average number of grains of oxides having a grain size of 10 μm or more in 10 fields of view and existing in the texture images of each field of view area of 1075 μm × 1433 μm was 74.3 and the average number density was 2.79/mm^2 in Example 7; which satisfied the range of the embodiment of the present invention. Meanwhile, the average number of grains was 11.5, and the average number density was 7.47/mm^2 in Comparative Example 7; and both values deviated from the range of the embodiment of the present invention.

As a result of evaluating this target based on the same sputtering test as Example 1, the number of particles having a diameter of 0.07 μm or more observed on the silicon substrate was 84 in Example 7 and 161 in Comparative Example 7; thus a significant difference was confirmed.

[0054] The foregoing results are shown in Table 1.

[Table 1]

	Component Composition of Sputtering Target (mol%)	Magnetic Material Raw Material Powder	Oxide Raw Material Powder	Heat-Treated Oxide Raw Material Powder	Heat Treatment Temperature	Heat Treatment Time	Sintering Temperature	Number Density of Oxide Particles of 10 μm or Larger (mm-2)	Number of Particles Having a Diameter of 0.07 μm or More
Example 1	70Co−4Cr-10Pt-4B_2O_3-2TiO_2−2SiO_2−2Cr_2O_3-6CoO	Co, Cr, Pt	B_2O_3, TiO_2, SiO_2, Cr_2O_3, CoO	TiO_2, SiO_2	1050°C	5 hours	850°C	1.88	51
Comparative Example 1	70Co−4Cr-10Pt-4B_2O_3-2TiO_2−2SiO_2−2Cr_2O_3-6CoO	Co, Cr, Pt	B_2O_3, TiO_2, SiO_2, Cr_2O_3, CoO	-	-	-	850°C	8.11	129
Example 2	65Co−20Pt-5B_2O_3-5TiO_2−5SiO_2	Co, Pt	B_2O_3, TiO_2, SiO_2	TiO_2, SiO_2	1050°C	5 hours	850°C	4.54	76
Comparative Example 2	65Co−20Pt-5B_2O_3-5TiO_2−5SiO_2	Co, Pt	B_2O_3, TiO_2, SiO_2	-	-	-	850°C	6.49	88
Example 3	65Co−20Cr-5B_2O_3-5TiO_2−5SiO_2	Co, Cr	B_2O_3, TiO_2, SiO_2	TiO_2, SiO_2	1050°C	5 hours	850°C	2.27	70
Comparative Example 3	65Co−20Cr-5B_2O_3-5TiO_2−5SiO_2	Co, Cr	B_2O_3, TiO_2, SiO_2	-	-	-	850°C	7.27	118
Example 4	65Co−20Cr-5B_2O_3-10TiO_2	Co, Cr	B_2O_3, TiO_2	B_2O_3, TiO_2	950°C	5 hours	850°C	4.67	98
Comparative Example 4	65Co−20Cr-5B_2O_3-10TiO_2	Co, Cr	B_2O_3, TiO_2	-	-	-	850°C	10.06	217
Example 5	65Co−20Cr-5B_2O_3-10SiO_2	Co, Cr	B_2O_3, SiO_2	B_2O_3, SiO_2	850°C	5 hours	850°C	3.31	66
Comparative Example 5	65Co−20Cr-5B_2O_3-10SiO_2	Co, Cr	B_2O_3, SiO_2	-	-	-	850°C	5.13	77
Example 6	65Co−20Cr-5B_2O_3-10Cr_2O_3	Co, Cr	B_2O_3, Cr_2O_3	B_2O_3, Cr_2O_3	850°C	5 hours	850°C	4.61	102
Comparative Example 6	65Co−20Cr-5B_2O_3-10Cr_2O_3	Co, Cr	B_2O_3, Cr_2O_3	-	-	-	850°C	9.28	182

Industrial Applicability

[0055] The present invention provides a magnetic sputtering target, with the texture thereof, capable of suppressing the aggregation caused particularly by the low-melting-point oxides; and thereby suppresssing the abnormal discharge and reducing the generation of particles caused by coarse oxides during sputtering. Consequently the magnetic sputtering target yields a superior effect of further cost improvement based on the improvement of the production yield, and is useful as a magnetic sputtering target used for depositing a magnetic thin film of a magnetic recording medium, particularly in depositing a hard disk drive recording layer.

CLAIMS

1. A magnetic sputtering target including oxides having a melting point of 500°C or lower, comprising; an average number density of oxides having a grain size of 10 μm or more on a sputter surface of the sputtering target is 5/mm^2 or less.

2. The magnetic sputtering target according to claim 1, wherein the sputtering target includes oxides has one or more types selected from Cr, Ta, Ti, Si, Zr, Al, Nb, and Co.

3. The magnetic sputtering target according to claim 1 or claim 2, wherein a total content of oxides in the sputtering target is 5 vol% to 50 vol%.

4. The magnetic sputtering target according to any one of claims 1 to 3, wherein Co is 55 mol% or more and 95 mol% or less, Cr is 40 mol% or less, and Pt is 45 mol% or less in the sputtering target.

5. The magnetic sputtering target according to claim 4, wherein the sputtering target includes 10 mol% or less of one or more types selected from B, N, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Si, and Al.

6. A production method of the magnetic sputtering target according to any one of claims 1 to 5, comprising; heat treating an oxide powder containing oxides having a melting point of 500°C or lower at a temperature that is equal to or higher than a sintering temperature of a target, and using the heat-treated powder as a sintering material.

7. A production method of the magnetic sputtering target according to any one of claims 1 to 5, comprising; heat treating an oxide powder excluding oxides having a melting point of 500°C or lower at a temperature that is equal to or higher than a sintering temperature of a target, and using the heat-treated powder as a sintering material.

8. The production method of a magnetic sputtering target according to claim 6 or claim 7 including a step of performing heat treatment in an atmosphere at 800°C or higher and 1900°C or lower.

9. The production method of a magnetic sputtering target according to claim 6 including a step of adjusting a grain size of the heat-treated oxide powder to be an average grain size of 5 μm or less.

10. The production method of a magnetic sputtering target according to any one of claims 6 to 9 including a step of performing hot press sintering at a holding temperature of 500°C to 1400°C.