

MAGNETIC MATERIAL SPUTTERING TARGET AND MANUFACTURING METHOD THEREOF

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BACKGROUND

[0001] The present invention relates to a ferromagnetic sputtering target used for depositing a thin film in a magnetic recording medium, in particular depositing a granular film on a magnetic recording medium for a hard disk in which the perpendicular magnetic recording system is used. The present invention also relates to a nonmagnetic grain-dispersed ferromagnetic sputtering target capable of suppressing discharge abnormalities of oxides that are the cause of particle generation during sputtering and a producing method thereof.

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[0002] Although a wide variety of systems are available for a sputtering device, a magnetron sputtering device equipped with a DC power supply is widely used in view of high productivity to deposit the above magnetic recording films. The sputtering method refers to one in which a substrate serving as a positive electrode is placed facing a target which serves as a negative electrode, and high voltage is applied between the substrate and the target under an inert gas atmosphere to generate an electric field.

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[0003] At this time, the inert gas is ionized to form plasma which comprises electrons and cations. When the cations in the plasma hit the surface of the target (the negative electrode), atoms which constitute the target are ejected. A film is formed when the ejected atoms adhere to the surface of the opposing substrate. The method employs the principle that a material which constitutes the target is deposited on the substrate by a series of these operations.

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[0004] Meanwhile, referring to the development of magnetic materials, in the field of magnetic recording represented by hard disk drives, materials based on ferromagnetic metals such as Co, Fe or Ni are used as materials for magnetic thin films which serve for recording. For example, in recording layers of hard disks in which the longitudinal magnetic recording system is used, Co-Cr based or Co-Cr-Pt based ferromagnetic alloys having Co as a main component are used.

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[0005] In addition, for recording layers of hard disks employing the perpendicular magnetic recording system which has been recently put in practical use, composite materials comprising a Co-Cr-Pt based ferromagnetic alloy having Co as a main component and a nonmagnetic inorganic substance

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are often used.

In many cases, a magnetic thin film in a magnetic recording medium such as a hard disk is produced by sputtering a ferromagnetic material sputtering target having the above materials as main components because of high productivity.

[0006] Methods of producing the foregoing ferromagnetic sputtering target include the dissolution method and the powder metallurgy method. Which methods may be used depends on required properties. But a sputtering target comprising a ferromagnetic alloy and grains of a nonmagnetic inorganic substance, which is used for recording layers for hard disks of the perpendicular magnetic recording system, is generally produced by the powder metallurgy method. This is because producing is difficult by the dissolution method since the grains of an inorganic substance need to be uniformly dispersed in the alloy base.

[0007] For example, a method of obtain a sputtering target for a magnetic recording medium is proposed, the method comprising: performing mechanical alloying on an alloy powder having a metal phase produced by the rapid solidification method and a powder for a ceramics phase to uniformly disperse the powder for a ceramics phase in the alloy powder; and performing molding by hot press (Patent Literature 1). In this case, the structure of the target shows an appearance where the base materials are connected in a form of soft roe (cod sperm), which are surrounded by SiO₂ (ceramics) (Fig. 2 of Patent Literature 1) or an appearance where the base materials are dispersed in a form of a thin string (Fig. 3 of Patent Literature 1). Other figures are assumed to show the similar structures although they are not clear. These structures have problems described below, and cannot be considered as a suitable sputtering target for a magnetic recording medium. Note that spherical materials shown in Fig. 4 of Patent Literature 1 are of powder, and are not the structure of the target.

[0008] Further, even in a case where an alloy powder produced by the rapid solidification method is not used, a ferromagnetic sputtering target can be produced by preparing commercially available raw powders for each component which will constitute the target, weighing out the raw powders to obtain a desired composition, mixing by a known method such as ball milling, and molding and sintering the mixed powder by hot press.

[0009] For example, a method of obtaining a sputtering target for a magnetic recording medium is proposed, the method comprising: mixing a mixed powder obtained by mixing a Co powder, a Cr powder, a TiO₂ powder and a SiO₂ powder,

with a Co spherical powder by a planetary mixer, molding the resulting powder mixture by hot press.

[0010] In this case, the structure of the target shows an appearance where there is a spherical phase (B) in a phase (A) in which grains of an inorganic substance are uniformly dispersed in the metal base (Fig. 1 of Patent Literature 2). The structure as described above is suitable in terms of improving magnetic leakage flux, while in terms of suppressing particle generation during sputtering, it can hardly be considered as a suitable sputtering target for a magnetic recording medium.

10 [0011] In addition, a method of obtaining a sputtering target for forming a magnetic recording medium thin film is proposed, wherein the method comprising: mixing a Co-Cr binary alloy powder, a Pt powder and a SiO₂ powder, and hot pressing the resulting mixed powder (Patent Literature 3).

[0012] In the structure of the target in this case, a Pt phase, a SiO₂ phase and a Co-Cr binary alloy phase were observed, and a dispersed layer was observed around a Co-Cr binary alloy layer although not shown in the figures. Such a structure as described above, it can hardly be considered as a suitable sputtering target for a magnetic recording medium.

[0013] Further to these described above, there are a few more proposals for developing a magnetic material. For example, a perpendicular magnetic recording medium having SiC and SiO_x (x: 1 to 2) is proposed in Patent Literature 4. Patent Literature 5 describes a magnetic material target containing Co, Pt, a first metal oxide, a second metal oxide and a third metal oxide.

[0014] Further, in Patent Literature 6, proposed is a sputtering target comprising a Co-Pt matrix phase and a metal oxide phase having an average grain diameter of 0.05 μm or more and less than 7.0 μm . Also proposed is that controlling growth of crystal grains to obtain a target having low magnetic permeability and high density, thereby increasing deposition efficiency.

Furthermore, Patent Literature 7 describes a nonmagnetic grain-dispersed ferromagnetic sputtering target in which a shape of a nonmagnetic material (smaller than a virtual circle having a radius of 2 μm) is defined with a material having ferromagnetic materials Co and Fe as main components and having a material selected from the group consisting of oxide, nitride, carbide and silicide.

35 [0015] Moreover, Patent Literature 8 describes a nonmagnetic grain-dispersed ferromagnetic sputtering target in which nonmagnetic material grains comprising, oxides smaller than a virtual circle having a radius of 1 μm are dispersed in a

Co-Cr alloy ferromagnetic material. It also describes a sputtering target in which the grain-diameter thereof is defined in detail. In addition, Patent Literature 9 describes a magnetic film having a granular structure.

[0016] As described above, for nonmagnetic grain-dispersed ferromagnetic sputtering targets such as a Co-Cr-Pt-oxide, use of SiO₂, Cr₂O₃ and TiO₂ as an oxide has been proposed, and defining a shape of the oxide has been further proposed. However, these oxides can cause abnormal electric discharge since they are insulators. Therefore, this abnormal electric discharge will pose a problem of particle generation during sputtering.

10 [0017] The amount of levitation of a magnetic recording head is becoming smaller every year as the recording density of HDD is improved. Therefore, the size and the number of particles permissible on magnetic recording medium are becoming even stricter. It is known that many of particles generated during the deposition of a granular film are oxides from the target. As one method of suppressing such particle generation, finely dispersing oxides in a target into a parent phase alloy appears to be effective.

[0018] In addition to Patent Literatures 6 to 8 described above, a smaller grain size of a metal oxide is also proposed in Patent Literatures 10 to 19 described below. That is, Patent Literature 10 describes that an average grain diameter of grains formed in a metal oxide phase is 0.05 μm or more and less than 7.0 μm . Patent Literature 11 describes that the grain diameter along the long axis of a ceramics phase is 10 μm or less. Patent Literature 12 describes that an oxygen-containing substance or an oxide phase is 50 μm or less. Patent Literature 13 describes that an average grain diameter of grains formed in an oxide phase is 3 μm or less. Patent Literature 14 describes that silica grains or titania grains satisfy $2 \leq D_p/D_n$ at a cross section perpendicular to the main surface of a sputtering target where D_n represents a grain diameter in the direction perpendicular to the main surface of the sputtering target, and D_p represents a grain diameter in the direction parallel to the above main surface. Patent Literature 15 describes that there are 500 chromium oxide aggregates present per mm^2

[0019] Moreover, Patent Literature 16 describes that in a Co based alloy sputtering target containing silica, Cr or Pt, wherein the silica phase is in the range 0.5 to 5 μm and a hydrophobic silica powder can be used for manufacture. Patent Literature 17 describes a sputtering target used for producing a magnetic recording medium, wherein the grain diameters of oxides are 10 μm or less. Patent Literature 18 describes a Co-Cr-Pt-C based sputtering target, wherein an

average crystal grain size in the matrix is 50 μm or less, and carbide is dispersed in the structure. Patent Literature 19 describes a magnetic recording medium wherein crystal grains constituting a magnetic thin film are separated from a crystal grain boundary portion comprising a non-ferromagnetic non-metal phase. However, none of these grain-micronizing conditions are sufficient, and the demand for further improvement still remains at the present.

[CITATION LIST]

[Patent Literature]

- 10 [0020] Patent Literature 1: Japanese Patent Laid-Open No. H10-88333
Patent Literature 2: Japanese Patent Application No. 2010-011326
Patent Literature 3: Japanese Patent Laid-Open No. 2009-1860
Patent Literature 4: Japanese Patent Laid-Open No. 2006-127621
Patent Literature 5: Japanese Patent Laid-Open No. 2007-4957
15 Patent Literature 6: Japanese Patent Laid-Open No. 2009-102707
Patent Literature 7: Domestic Re-publication of WO2007/080781
Patent Literature 8: WO2009/119812A1
Patent Literature 9: Japanese Patent Laid-Open No. 2001-76329
Patent Literature 10: WO2009/054369
20 Patent Literature 11: Japanese Patent Laid-Open No. 2006-045587
Patent Literature 12: Japanese Patent Laid-Open No. 2008-169464
Patent Literature 13: Japanese Patent Laid-Open No. 2009-215617
Patent Literature 14: Japanese Patent Laid-Open No. 2011-222086
Patent Literature 15: Japanese Patent Laid-Open No. 2008-240011
25 Patent Literature 16: Japanese Patent Laid-Open No. 2004-339586
Patent Literature 17: Japanese Patent Laid-Open No. 2001-236643
Patent Literature 18: Japanese Patent Laid-Open No. 2001-98360
Patent Literature 19: Japanese Patent Laid-Open No. H7-311929

SUMMARY OF INVENTION

[TECHNICAL PROBLEM]

[0021] In general, since nonmagnetic grain-dispersed ferromagnetic sputtering targets such as a Co-Cr-Pt oxide includes oxides therein such as SiO_2 , Cr_2O_3 , TiO_2 , Ta_2O_5 , CoO , Co_3O_4 and B_2O_3 which are insulators, the oxides may become a cause for abnormal electric discharge. Therefore, this abnormal electric discharge will pose a problem of particle generation during sputtering.

In view of the above problem, the present invention aims to suppress abnormal electric discharge due to oxides and to reduce particle generation caused by abnormal electric discharge during sputtering. Although previous attempts to reduce the probability of abnormal electric discharge have been made by decreasing the grain diameter of oxides, a permissible particle level is becoming stricter as the recording density of a magnetic recording medium is improved. Accordingly, an object of the present invention is to provide a nonmagnetic grain-dispersed ferromagnetic sputtering target in which the dispersed state of oxides is further improved.

[SOLUTION TO PROBLEM]

[0022] After conducting extensive studies in order to achieve the above objects, the present inventors find that a target in which abnormal electric discharge due to oxides does not occur during sputtering and particle generation is reduced can be obtained by adjusting the structure (oxide grain) of the target.

[0023] Based on these findings, the present invention provides:

1) An oxide-containing magnetic material sputtering target, wherein the oxides have an average grain diameter of 400 nm or less.

2) The magnetic material sputtering target according to 1) above, comprising Phase A in which the oxides have an average grain diameter of 400 nm or less, and Phase B in which the oxides surrounding Phase A have an average grain diameter of 2 μ m or less.

3) The oxide-containing magnetic material sputtering target according to any one of 1) to 2) above, having an oxide structure in which an average grain diameter is 400 nm or less and an aspect ratio is 2 or less.

4) The oxide-containing magnetic material sputtering target according to any one of 1) to 3) above, comprising Phase A having an oxide structure in which an average grain diameter is 400 nm or less and the aspect ratio is 2 or less for a rectangular circumscribing an oxide grain to have an area thereof at the minimum, and Phase B in which the oxides surrounding Phase A have an average grain diameter of 2 μ m or less.

[0024] 5) The magnetic material sputtering target according to any one of 1) to 4) above, wherein Cr is 45 mol% or less (except 0 mol%) and Co.

6) The magnetic material sputtering target according to any one of above 1) to 4) above, wherein Cr is 45 mol% or less (except 0 mol%), Pt is 1 mol% or more and 30 mol% or less and Co.

7) The magnetic material sputtering target according to any one of 1) to 4) above, wherein Pt is 1 mol% or more and 30 mol% or less and Co.

8) The magnetic material sputtering target according to any one of 1) to 4) above, wherein Pt is 5 mol% or more and 60 mol% or less and Fe.

[0025] 9) The magnetic material sputtering target according to any one of 1) to 8) above, wherein as an additive element, one or more elements selected from B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Ag, Au, Cu and C in an amount of 0.5 mol% or more and 20 mol% or less is added.

10) The magnetic material sputtering target according to any one of 1) to 9) above, wherein as an oxide raw material, an oxide of one or more components selected from B, Si, Cr, Ti, Ta, W, Al, Mg, Mn, Ca, Zr, and Y in an amount of 1 to 20 mol% is added.

11) The magnetic material sputtering target according to any one of 1) to 10) above, wherein as an additive material, an inorganic substance material having one or more components selected from carbon, nitride and carbide is added.

12) The magnetic material sputtering target according to any one of 1) to 11) above, wherein HIP compaction treatment is performed after hot pressing.

[0026] 13) A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, and further sintering the raw material.

14) The method of producing the oxide-containing magnetic material sputtering target according to any one of 1) to 12) above, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, and further sintering the raw material.

25 [0027] 15) A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, further mixing the raw material with added insufficient components, and sintering the mixture.

30 16) The method of producing the oxide-containing magnetic material sputtering target according to any one of 1) to 12) above, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, further mixing the raw material with added insufficient components, and sintering the mixture.

[0028] 17) A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD,

removing the substrate from the deposited magnetic material, and further performing hot isostatic pressing (HIP) on the resulting magnetic material.

18) The method of producing the oxide-containing magnetic material sputtering target according to any one of 1) to 12) above, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, and further performing hot isostatic pressing (HIP) on the resulting magnetic material.

[0029] 19) A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD, and removing the substrate from the deposited magnetic material to obtain a target.

20) The method of producing the oxide-containing magnetic material sputtering target according to any one of 1) to 12) above, by depositing a magnetic material on a substrate by PVD or CVD, and removing the substrate from the deposited magnetic material to obtain a target.

21) The method of producing an oxide-containing magnetic material sputtering target according to any one of 17) to 20) above, by removing the substrate from the deposited magnetic material, and laminating the obtained thin film to obtain a target.

[EFFECTS OF INVENTION]

[0030] According to the present invention, the structure of a ferromagnetic sputtering target, in particular the shape of oxide grains can be adjusted (micronized) to reduce contamination of impurities from a grinding device and a medium. Therefore, abnormal electric discharge due to oxides during sputtering will not occur, and particle generation can be reduced. Therefore, when the target of the present invention is used, stable electric discharge can be obtained during sputtering using magnetron sputtering equipment. Further, the following advantageous effects can be achieved: abnormal electric discharge due to oxides can be suppressed; particle generation during sputtering caused by abnormal electric discharge can be reduced; and cost improvement can be achieved by improved yields.

BRIEF DESCRIPTION OF DRAWINGS

[0031] Fig. 1 shows images (photographs) illustrating the structure of the Co-Pt-Cr-SiO₂-TiO₂-Cr₂O₃ based target in Example 1.

Fig. 2 shows an image (photograph) illustrating the structure of the

Co-Pt-Cr-SiO₂-TiO₂-Cr₂O₃ based target in Comparative Example 1.

Fig. 3 shows images (photographs) illustrating the structure of the Co-Pt-Ru-Cr-SiO₂-TiO₂-CoO based target in Example 2.

Fig. 4 shows an image (photograph) illustrating the structure of the Co-Pt-Ru-Cr-SiO₂-TiO₂-CoO based target in Comparative Example 2.

Fig. 5 shows images (photographs) illustrating the structure of the Co-Cr-TiO₂ based target in Example 5.

Fig. 6 shows images (photographs) illustrating the structure of the Co-Pt-SiO₂-TiO₂-Cr₂O₃ based target in Example 6.

Fig. 7 shows an image (photograph) illustrating that Phase B of relatively large oxides is present at the grain boundary of Phase A of oxides in the Co-Pt-Ru-Cr-SiO₂-TiO₂-CoO based target in Example 2.

Fig. 8 shows an explanatory image illustrating that Phase B of relatively large oxides is present at the grain boundary of Phase A of oxides in the Co-Pt-Ru-Cr-SiO₂-TiO₂-CoO based target in Example 2.

DETAILED DESCRIPTION

[0032]

As the storage capacity of a hard disk drive is increasing, there has been a demand to reduce particles from a sputtering target when producing a hard disk medium in recent years. A sputtering target for perpendicular magnetic recording is commonly produced by powder sintering. In order to reduce particles, micronizing the structure of a sputtering target is very effective.

A sputtering target for perpendicular magnetic recording comprises a ferromagnetic material and a nonmetal material such as oxides and carbon. In order to suppress particles during sputtering, the metal and nonmetal need to be finely and uniformly dispersed.

In order to achieve this, mechanically pulverizing and mixing raw powders together using a powerful ball mill and the like is one of the effective approaches. However, there are physical limitations in micronizing the structure by the current mechanical pulverization and mixing approaches, and therefore particle generation cannot be completely eliminated.

[0033]

Accordingly, an ultrafine structure is achieved herein by using the vapor deposition methods instead of the conventional mechanical pulverization and mixing. The vapor deposition methods can include the PVD method (Physical Vapor Deposition) or the CVD method (Chemical Vapor Deposition).

In general, PVD and CVD are approaches for producing a thin film. In

principle, these approaches involve decomposing a material to a molecular level, and then reconstituting it to form a thin film. Therefore the thin film will have a structure which is far finer than that from mechanical pulverization and mixing. Therefore, the above problem can be solved by producing a sputtering target from a film obtained by the PVD and CVD approaches.

[0034] The oxide-containing magnetic material sputtering target of the present invention can be produced by depositing a magnetic material on a substrate by PVD or CVD, then removing the substrate from the deposited magnetic material, then pulverizing the resulting material to give a raw material for the target, and further sintering the raw material. Removing a substrate from a thin film which is deposited on the substrate in particular by the sputtering method or the vapor deposition method among PVD or CVD is effective to obtain a sputtering material.

[0035] They can be characterized by easiness of producing because they can be used for deposition in large-sized equipment, and can also produce a thickness of about 1000 μm . They have the following advantages: attendance is not required during deposition; further, the resulting sputtering material is almost free from contamination; and little contamination is introduced during pulverization because mechanical pulverization can be easily performed.

Further when sintering, HIP compaction treatment after hot pressing is effective, but sintering conditions are not limited to this, and any can be used.

[0036] In some cases, a material pre-deposited on a substrate by PVD or CVD may not be consistent with a component composition of a magnetic material sputtering target to be produced. In this case, an oxide-containing magnetic material sputtering target can also be produced by pre-depositing a magnetic material having a similar component composition on a substrate, then removing the substrate from the deposited magnetic material, pulverizing the resulting material to give a raw material for the target, further adding an insufficient component to the raw material, and sintering the mixture.

[0037] The removal of the substrate after deposition as described above can be performed by mechanical removal or chemical dissolution removal or in appropriate combination thereof, but there is no particular limitation. Preferably, contamination of the sintering raw material with the substrate after removing the substrate can be controllable. Further, contamination with impurities can be significantly reduced even when a substrate material is similar or the same to a sintering material is used.

As a supplemental material to add insufficient materials, preferably fine

grains similar to the sintering raw material are used; however, since they will account for a small amount of the sintering raw materials, it will not get much influence

[0038] Upon sintering a supplemental material once pulverization is performed after recovering a vapor deposited film, a structure in which oxides larger than those within the vapor deposited film are dispersed may be seen at the grain boundary between the pulverized powders of the vapor deposited film.

5 However, the presence of the oxides at the grain boundary of the vapor deposited film will not become a crucial problem, since the oxides at the grain boundary are small enough as compared with those mechanically pulverized and mixed though they are larger than those in the vapor deposited film; in addition, most of the sintered compact shows a fine structure such that these large grains will give not much influence.

[0039] While the above-mentioned method is steps in that magnetic material is deposited on a substrate by PVD or CVD, then the substrate is removed from the deposited magnetic material, and the resulting material is pulverized to obtain a raw material, it is also possible that hot isostatic pressing (HIP) may be performed on the thin film itself.

20 Further, the substrate can be removed from the deposited magnetic material to obtain a target as it is. Furthermore, the substrate is removed from the deposited magnetic material, and the resulting thin films are laminated, which can be then subjected to press processing such as hot isostatic pressing (HIP) to give an oxide-containing magnetic material sputtering target.

25 The thin film and the laminated structure obtained as described above may have any thickness and any number of layers, respectively, which can be suitably determined depending on the material and the requirement. Further, an insufficient component may be added, and sintering can be performed to produce an oxide-containing magnetic material sputtering target. The present invention shall encompass all of them.

30 [0040] Thus, an oxide-containing magnetic material sputtering target can be obtained, which has an average grain diameter of 400 nm or less. This is a basic structure of the target which can be achieved by the present invention.

35 It is also possible to obtain an oxide-containing magnetic material sputtering target, wherein the oxide-containing magnetic material sputtering target comprising Phase A in which the oxides have an average grain diameter of 400 nm or less and Phase B in which the oxides surrounding Phase A have an average grain diameter of 2 μ m or less; further an oxide-containing magnetic

material sputtering target having an oxide structure in which an average grain diameter is 400 nm or less, and the aspect ratio is 2 or less; and an oxide-containing magnetic material sputtering target comprising Phase A having an oxide structure in which an average grain diameter is 400 nm or less and the aspect ratio is 2 or less for a rectangular circumscribing an oxide grain to have an area thereof at the minimum and Phase B in which the oxides surrounding Phase A have an average grain diameter of 2 μ m or less.

[0041] The above magnetic material sputtering targets may be a magnetic material sputtering target in which Cr is less than 45 mol% (except 0 mol%) and Co; a magnetic material sputtering target in which Cr is less than 45 mol% (except 0 mol%), and Pt is 1 mol% or more and 30 mol% or less and Co; and a magnetic material sputtering target in which Pt is 5 mol% or more and 60 mol% or less and Fe.

[0042] Further, the followings can be obtained: the above magnetic material sputtering target containing one or more elements selected from B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Ag, Au, Cu and C in an amount of 0.5 mol% or more and 20 mol% or less as an additive element; the above magnetic material sputtering target containing an oxide of one or more components selected from B, Si, Cr, Ti, Ta, W, Al, Mg, Mn, Ca, Zr and Y in an amount of 1 to 20 mol% as an oxide raw material; the above magnetic material sputtering target containing an inorganic material having one or more components selected from carbon, nitride and carbide as an additive material; and a magnetic material sputtering target in which HIP compaction treatment is performed after hot pressing.

[0043] The amounts of the above elements to be included merely show suitable numerical ranges in order to take advantage of the properties of a ferromagnetic material. Needless to say, any values which are not listed above can be used if desired. The present invention shall encompass all of them. The above amount of addition is effective amounts to show an effect of addition.

The resulting raw materials for sintering are weighed out to give a desired composition, and mixed and pulverized using a known approach such as a mortar and a ball mill. Raw powders to add as insufficient materials may be mixed at this stage. Further, there is no particular limitation for pulverizers and mixers, but a planetary mixer or a planetary stirring mixer can be preferably used. Moreover, when considering an issue of oxidation during mixing, mixing is preferably performed under an inert gas atmosphere or in vacuum.

[0044] In the present specification, described are Cr-Co alloy based magnetic materials, Cr-Pt-Co alloy based magnetic materials, Pt-Co alloy based magnetic

materials, Pt-Fe alloy based magnetic materials which are typical as oxide-containing magnetic materials. The present invention is characterized by the shape of oxides present in a magnetic material and a producing method to obtain these special oxides (the form of presence).

Therefore, it should be easily understood that the present invention can be similarly applied to other component systems such as Fe-Ni alloy based magnetic materials, Fe-Co alloy based magnetic materials, Fe-Ni-Co alloy based magnetic materials to obtain a similar effect. The present invention shall encompass all of them.

EXAMPLES

The Examples are now explained based on Examples and Comparative Examples. Note that these Examples are merely illustrative, and the present invention shall in no way be limited thereby. In other words, various modifications and other embodiments are covered by the present invention, and the present invention is limited only by the scope of its claims.

(Example 1)

A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a TiO_2 powder having an average grain diameter of 1 μm , a SiO_2 powder having an average grain diameter of 0.7 μm and a Cr_2O_3 powder having an average grain diameter of 1 μm were prepared as oxide powders. These powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 70Co-18Pt-3Cr-4 SiO_2 -2 TiO_2 -3 Cr_2O_3 (mol%)

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 resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was

performed at 2 kWhr; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 4000 μm .

Then, the substrate was exfoliated from the sputter-deposited material, and hot isostatic pressing (HIP) was performed. The conditions of hot isostatic pressing were a rate of temperature increase at 300°C/h, a retention temperature of 950°C and a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 950°C. After retention ended, it was kept in the furnace to allow natural cooling.

[0049] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. Structure images of this target are shown in Fig. 1; and as shown in the figure, obtained is a Co-Pt-Cr-SiO₂-TiO₂-Cr₂O₃ based target having fine oxide grains with an average grain diameter of 60 nm.

Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds.

[0050] The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions. The average number of particles on the Si substrate in this case was 0.3 having a particle size of 0.17 μm to 0.25 μm , and 0 having a particle size of 0.25 to 3.0 μm .

The results are shown in Table 1. A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

[0051] [Table 1]

	Composition	Hot Press Temperature (°C)	Grain Diameter after Film Pulverization (μm)	Film/Pulverized Powder Hot Press Temperature (°C)	HIP Temperature (°C)	Grain Diameter of Oxide (nm)	Particle 0.17 to 0.25 (μm)	Particle 0.25 to 3 (μm)
Example 1	70Co-18Pt-3Cr-4SiO ₂ -2TiO ₂ -3Cr ₂ O ₃ (mol%)	1050	—	—	950	60	0.3	0
Example 2	59Co-20Pt-5Ru-3Cr-5SiO ₂ -2TiO ₂ -6CoO (mol%)	1050	200	1000	1000	40	0.7	0.3
Example 3	76Co-12Pt-5Cr-5Ta ₂ O ₅ -2SiO ₂ (mol%)	1050	100	1000	1000	40	0.3	0.3
Example 4	70Co-10Pt-12Cr-2B ₂ O ₃ -3TiO ₂ -2SiO ₂ -1CoO (mol%)	950	250	950	950	60	1	0.7
Example 5	50Co-40Cr-10TiO ₂ (mol%)	950	50	950	950	70	0.3	0
Example 6	72Co-20Pt-3SiO ₂ -2TiO ₂ -3Cr ₂ O ₃ (mol%)	1050	—	—	1100	400	3	1.3
Example 7	85Co-5Pt-3Ru-2SiO ₂ -2TiO ₂ -3Cr ₂ O ₃ (mol%)	1050	300	1000	1000	350	4	2
Example 8	67.5Co-10Pt-12Cr-3B-6SiO ₂ -1.5Cr ₂ O ₃	900	250	850	850	150	1.3	1
Example 9	79Fe-5Pt-16SiO ₂ (mol%)	1090	150	1000	1000	90	1	0.3
Example 10	29Fe-55Pt-16SiO ₂ (mol%)	1090	150	1000	1000	80	1.3	0.7
Example 11	70Co-12Pt-12Cr-3SiO ₂ -2TiO ₂ -1Cr ₂ O ₃ (mol%)	1050	—	1000	1000	60	0.3	0.3
Example 12	79Co-10Pt-6Cr-1Ta ₂ O ₅ -4SiO ₂ (mol%)	1050	200	950	950	10	0.3	0
Comparative Example 1	70Co-18Pt-3Cr-4SiO ₂ -2TiO ₂ -3Cr ₂ O ₃ (mol%)	1050	—	—	—	1300	10.7	5.3
Comparative Example 2	59Co-20Pt-5Ru-3Cr-5SiO ₂ -2TiO ₂ -6CoO (mol%)	1050	—	—	—	1600	12.3	7.3
Comparative Example 3	76Co-12Pt-5Cr-5Ta ₂ O ₅ -2SiO ₂ (mol%)	1050	—	—	—	1000	12	4.7
Comparative Example 4	70Co-10Pt-12Cr-2B ₂ O ₃ -3TiO ₂ -2SiO ₂ -1CoO (mol%)	950	—	—	—	1800	14.3	3.3
Comparative Example 5	50Co-40Cr-10TiO ₂ (mol%)	950	—	—	—	2300	8.7	3

5 (Comparative Example 1)

[0052] A Co powder having an average grain diameter of 4 μm, a Cr powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a TiO₂ powder having an average grain diameter of 1 μm, a SiO₂ powder having an average grain diameter of 0.7 μm and a Cr₂O₃ powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 70Co-18Pt-3Cr-4SiO₂-2TiO₂-3Cr₂O₃ (mol%)

15 [0053] The weighed powders were sealed in a 10-litter ball mill pot along with tungsten alloy balls as grinding media, and rotated for 100 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The

sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm.

[0054] When the structure of this target was observed, it showed that an average grain diameter of oxide grains within a microscopic field was 1.3 μm .
5 The structure of this target is shown in Fig. 2.

Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions. The average number of particles on the Si substrate in this case was 10.7 having a particle size of 0.17 μm to 0.25 μm , and 5.3 having a particle size of 0.25 to 3.0 μm , which showed an increase as compared with Example 1. The results are shown in Table 1.
15 (Example 2)

[0055] A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm , a Pt powder having an average grain diameter of 3 μm and a Ru powder having an average grain diameter of 5 μm
20 were prepared as metal raw powders; and a TiO_2 powder having an average grain diameter of 1 μm , a SiO_2 powder having an average grain diameter of 0.7 μm and a CoO powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

25 Composition: 59Co-20Pt-5Ru-3Cr-5SiO₂-2TiO₂-6CoO (mol%)

[0056] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention
30 time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0057] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .
35

Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 200 μm . The steps of sputtering to pulverization and recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1000°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1000°C, a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention temperature of 1000°C. After retention ended, it was kept in the furnace to allow natural cooling

[0058] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. Structure images of this target are shown in Fig. 3. As shown in Fig. 3, a Co-Pt-Ru-Cr-SiO₂-TiO₂-CoO based target having fine oxide grains with an average grain diameter of 40 nm was obtained.

For reference, Fig. 7 (photograph) shows that Phase B of relatively large oxides is present at the grain boundary of Phase A of oxides. In addition, Fig. 8 shows an explanatory image. Note that Fig. 7 and Fig. 8 each show the structures observed at different places.

Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0059] The average number of particles on the Si substrate in this case was 0.7 having a particle size of 0.17 μm to 0.25 μm , and 0.3 having a particle size of 0.25 to 3.0 μm .

The results are shown in Table 1. A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering

equipment.

(Comparative Example 2)

[0060] A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm , a Pt powder having an average grain diameter of 3 μm and a Ru powder having an average grain diameter of 5 μm were prepared as metal raw powders, and a TiO_2 powder having an average grain diameter of 1 μm , a SiO_2 powder having an average grain diameter of 0.7 μm and a CoO powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 59Co-20Pt-5Ru-3Cr-5 SiO_2 -2 TiO_2 -6CoO (mol%)

[0061] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 100 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm.

When the structure of this target was observed, it showed that an average grain diameter of oxide grains within a microscopic field was 1.6 μm . The structure of this target is shown in Fig. 4.

[0062] Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

The average number of particles on the Si substrate in this case was 12.3 having a particle size of 0.17 μm to 0.25 μm , and 7.3 having a particle size of 0.25 to 3.0 μm , which showed an increase as compared with Example 2. The results are shown in Table 1.

(Example 3)

[0063] A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a Ta_2O_5

powder having an average grain diameter of 1 μm and a SiO_2 powder having an average grain diameter of 0.7 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

5 Composition: 76Co-12Pt-5Cr-5Ta₂O₅-2SiO₂ (mol%)

[0064] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0065] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

15 Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 100 μm . The steps of sputtering to pulverization and recovery were repeated for 4 times. The fine powder was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1000°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

20 Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1000°C, a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention temperature of 1000°C. After retention ended, it was kept in the furnace to allow natural cooling.

25 The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. A Co-Pt-Cr-Ta₂O₅-SiO₂ based target having fine oxide grains with an average grain diameter of 40 nm.

[0066] Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering

conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0067] The average number of particles on the Si substrate in this case was 0.3 having a particle size of 0.17 μm to 0.25 μm , and 0.3 having a particle size of 0.25 to 3.0 μm . The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

(Comparative Example 3)

[0068] A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a Ta_2O_5 powder having an average grain diameter of 1 μm and a SiO_2 powder having an average grain diameter of 0.7 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 76Co-12Pt-5Cr-5 Ta_2O_5 -2 SiO_2 (mol%)

[0069] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 100 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm.

When the structure of this target was observed, it showed that an average grain diameter of oxide grains within a microscopic field was 1 μm .

[0070] Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured

with a particle counter, which was repeated 3 times in the same sputtering conditions. The average number of particles on the Si substrate in this case was 12 having a particle size of 0.17 μm to 0.25 μm , and 4.7 having a particle size of 0.25 to 3.0 μm , which showed an increase as compared with Example 3. The results are shown in Table 1.

(Example 4)

[0071] A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a B_2O_3 powder having an average grain diameter of 10 μm , a TiO_2 powder having an average grain diameter of 1 μm , a SiO_2 powder having an average grain diameter of 0.7 μm and a CoO powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 70Co-10Pt-12Cr-2 B_2O_3 -3 TiO_2 -2 SiO_2 -1CoO (mol%)

[0072] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 950°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0073] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1.2 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 250 μm . The steps of sputtering to pulverization and recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 950°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 950°C and a retention time of 2 hours, and the gas

pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 950°C. After retention ended, it was kept in the furnace to allow natural cooling.

[0074] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. A Co-10Pt-12Cr-2B₂O₃-3TiO₂-2SiO₂-1CoO based target having fine oxide grains with an average grain diameter of 60 nm was obtained.

Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW/hr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0075] The average number of particles on the Si substrate in this case was 1 having a particle size of 0.17 μm to 0.25 μm, and 0.7 having a particle size of 0.25 to 3.0 μm. The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

(Comparative Example 4)

[0076] A Co powder having an average grain diameter of 4 μm, a Cr powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a B₂O₃ powder having an average grain diameter of 10 μm, a TiO₂ powder having an average grain diameter of 2 μm, a SiO₂ powder having an average grain diameter of 0.7 μm and a CoO powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 70Co-10Pt-12Cr-2B₂O₃-3TiO₂-2SiO₂-1CoO (mol%)

[0077] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 100 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 950°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The

sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm.

When the structure of this target was observed, it showed that an average grain diameter of oxide grains within a microscopic field was 1.8 μm .

5 [0078] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions. The average number of particles on the Si substrate in this case was 14.3 having a particle size of 0.17 μm to 0.25 μm , and 3.3 having a particle size of 0.25 to 3.0 μm , which showed an increase as compared with Example 4. The results are shown in Table 1.

15 (Example 5)

[0079] A Co powder having an average grain diameter of 4 μm and a Cr powder having an average grain diameter of 5 μm were prepared as metal raw powders, and a TiO_2 powder having an average grain diameter of 1 μm was prepared as an oxide powder. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 50Co-40Cr-10 TiO_2 (mol%)

[0080] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 950°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

30 [0081] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, sputtering was performed on a SUS substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

35 Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 50 μm . The steps of sputtering to pulverization and

recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 950°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

5 Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 950°C and a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 950°C. After
10 retention ended, it was kept in the furnace to allow natural cooling.

[0082] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. Structure images of this target are shown in Fig. 5. As shown in Fig. 5, a Co-40Cr-10TiO₂ based target having fine oxide grains with an average grain
15 diameter of 70 nm was obtained.

Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW/hr; and thereafter, deposition was
20 performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0083] The average number of particles on the Si substrate in this case was 0.3
25 having a particle size of 0.17 μm to 0.25 μm, and 0 having a particle size of 0.25 to 3.0 μm. The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when
30 sputtering was performed with magnetron sputtering equipment.

(Comparative Example 5)

[0084] A Co powder having an average grain diameter of 4 μm and a Cr powder having an average grain diameter of 5 μm were prepared as metal raw powders, and a TiO₂ powder having an average grain diameter of 1 μm was prepared as
35 an oxide powder. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 50Co-40Cr-10TiO₂ (mol%)

[0085] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 100 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 950°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm.

When the structure of this target was observed, it showed that an average grain diameter of oxide grains within a microscopic field was 2.3 μm .

10 [0086] Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW/hr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions. The average number of particles on the Si substrate in this case was 8.7 having a particle size of 0.17 μm to 0.25 μm , and 3 having a particle size of 0.25 to 3.0 μm , which showed an increase as compared with Example 5. The results are shown in Table 1.

(Example 6)

[0087] A Co powder having an average grain diameter of 4 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a SiO_2 powder having an average grain diameter of 0.7 μm , a TiO_2 powder having an average grain diameter of 1 μm , and a Cr_2O_3 powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

30 Composition: 72Co-20Pt-3SiO₂-2TiO₂-3Cr₂O₃ (mol%)

[0088] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0089] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 4000 μm .

5 Then, the substrate was exfoliated from the sputter-deposited material, and hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1100°C and a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 1100°C. After retention ended, it was kept in the furnace to allow natural cooling.

[0090] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. Structure images of this target are shown in Fig. 6. As shown in Fig. 6, a Co-Pt-SiO₂-TiO₂-Cr₂O₃ based target having fine oxide grains with an average grain diameter of 400 nm was obtained.

Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0091] The average number of particles on the Si substrate in this case was 3 having a particle size of 0.17 μm to 0.25 μm , and 1.3 having a particle size of 0.25 to 3.0 μm . The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment. (Example 7)

[0092] A Co powder having an average grain diameter of 4 μm , a Pt powder having an average grain diameter of 3 μm and a Ru powder having an average grain diameter of 5 μm were prepared as metal raw powders, and a SiO₂ powder having an average grain diameter of 0.7 μm , a TiO₂ powder having an average

grain diameter of 1 μm and a Cr_2O_3 powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

5 Composition: 85Co-5Pt-3Ru-2SiO₂-2TiO₂-3Cr₂O₃ (mol%)

[0093] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0094] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

15 Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 300 μm . The steps of sputtering to pulverization and recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1000°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

20 Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1000°C, a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 1100°C. After retention ended, it was kept in the furnace to allow natural cooling.

25 Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1000°C, a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 1100°C. After retention ended, it was kept in the furnace to allow natural cooling.

[0095] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. A Co-Pt-Ru-SiO₂-TiO₂-Cr₂O₃ based target having fine oxide grains with an average grain diameter of 350 nm was obtained.

30 Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering

conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0096] The average number of particles on the Si substrate in this case was 4 having a particle size of 0.17 μm to 0.25 μm , and 2 having a particle size of 0.25 to 3.0 μm . The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

(Example 8)

[0097] A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm , a Pt powder having an average grain diameter of 3 μm and a Co-B powder having an average grain diameter of 10 μm were prepared as metal raw powders, and a SiO_2 powder having an average grain diameter of 0.7 μm and a Cr_2O_3 powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 67.5Co-10Pt-12Cr-3B-6 SiO_2 -1.5 Cr_2O_3 (mol%)

[0098] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 900°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0099] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 250 μm . The steps of sputtering to pulverization and

recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 850°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

5 Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 850°C and a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 850°C. After
10 retention ended, it was kept in the furnace to allow natural cooling.

[0100] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. A Co-Pt-Cr-B-SiO₂-Cr₂O₃ target having fine oxide grains with an average grain diameter of 150 nm was obtained.

15 Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW/hr; and thereafter, deposition was
20 performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0101] The average number of particles on the Si substrate in this case was 1.3 having a particle size of 0.17 μm to 0.25 μm, and 1 having a particle size of 0.25
25 to 3.0 μm. The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

30 (Example 9)

[0102] An Fe powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a SiO₂ powder having an average grain diameter of 0.7 μm was prepared as an oxide powder. The powders were weighed at the following
35 composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 79Fe-5Pt-16SiO₂ (mol%)

[0103] The weighed powders were sealed in a 10-liter ball mill pot along with

zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1090°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0104] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 150 μm . The steps of sputtering to pulverization and recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1000°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1000°C, a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention temperature of 1000°C. After retention ended, it was kept in the furnace to allow natural cooling.

[0105] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. An Fe-Pt-SiO₂ based target having fine oxide grains with an average grain diameter of 90 nm was obtained.

Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0106] The average number of particles on the Si substrate in this case was 1

having a particle size of 0.17 μm to 0.25 μm , and 0.3 having a particle size of 0.25 to 3.0 μm . The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

(Example 10)

[0107] An Fe powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a SiO_2 powder having an average grain diameter of 0.7 μm was prepared as an oxide powder. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 29Fe-55Pt-16 SiO_2 (mol%)

[0108] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1090°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0109] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWh; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 150 μm . The steps of sputtering to pulverization and recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1000°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1000°C, a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention temperature of 1000°C.

After retention ended, it was kept in the furnace to allow natural cooling.

[0110] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. An Fe-Pt-SiO₂ based target having fine oxide grains with an average grain diameter of 80 nm was obtained.

5 Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW/hr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0111] The average number of particles on the Si substrate in this case was 1.3 having a particle size of 0.17 μm to 0.25 μm, and 0.7 having a particle size of 0.25 to 3.0 μm. The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment. (Example 11)

[0112] A Co powder having an average grain diameter of 4 μm, a Pt powder having an average grain diameter of 3 μm and a Cr powder having an average grain diameter of 5 μm were prepared as metal raw powders, and a SiO₂ powder having an average grain diameter of 0.7 μm, a TiO₂ powder having an average grain diameter of 1 μm and a Cr₂O₃ powder having an average grain diameter of 1 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

30 Composition: 70Co-12Pt-12Cr-3SiO₂-2TiO₂-1Cr₂O₃ (mol%)

[0113] The weighed powders were sealed in a 10-liter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0114] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW/hr; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 5 to 1000 μm .

5 Then, the substrate was exfoliated from the sputter-deposited material to recover a film. The steps from sputtering deposition to film recovery were repeated until the total thickness of the recovered films became 4000 μm . The recovered sheet-like thin films were laminated in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1000°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

[0115] Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 1000°C, a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention temperature of 1000°C. After retention ended, it was kept in the furnace to allow natural cooling.

[0116] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. A Co-Pt-Cr-SiO₂-TiO₂-Cr₂O₃ based target having fine oxide grains with an average grain diameter of 60 nm was obtained.

20 Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW/hr; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

[0117] The average number of particles on the Si substrate in this case was 0.3 having a particle size of 0.17 μm to 0.25 μm , and 0.3 having a particle size of 0.25 to 3.0 μm . The results are shown in Table 1.

35 A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

(Example 12)

[0118] A Co powder having an average grain diameter of 4 μm , a Cr powder having an average grain diameter of 5 μm and a Pt powder having an average grain diameter of 3 μm were prepared as metal raw powders, and a Ta_2O_5 powder having an average grain diameter of 1 μm and a SiO_2 powder having an average grain diameter of 0.7 μm were prepared as oxide powders. The powders were weighed at the following composition ratio to achieve 2000 g. The component composition is as follows.

Composition: 79Co-10Pt-6Cr-1Ta₂O₅-4SiO₂ (mol%)

10 [0119] The weighed powders were sealed in a 10-litter ball mill pot along with zirconia balls as grinding media, and rotated for 120 hours for mixing. The resulting mixture was filled in a carbon mold, and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 1050°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact. The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 180 mm and a thickness of 6 mm.

[0120] Subsequently, the target was placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kWhr; and thereafter, sputtering was performed on an Al substrate having a diameter of 6.5 inches to obtain a target thickness of 1000 μm .

20 Then, the substrate was exfoliated from the sputter-deposited material to recover a film, and the film was pulverized to obtain a fine powder having an average grain diameter of 200 μm . The steps of sputtering to pulverization and recovery were repeated for 4 times. The fine powder was filled in a carbon mold and hot pressed in a vacuum atmosphere under the following conditions: a temperature of 950°C, a retention time of 2 hours, and welding force of 30 MPa to obtain a sintered compact.

25 Then, hot isostatic pressing (HIP) was performed. The conditions for hot isostatic pressing were as follows: a rate of temperature increase at 300°C/h, a retention temperature of 950°C and a retention time of 2 hours, and the gas pressure of Ar gas was gradually increased from the time of starting temperature increase, and pressurized at 150 MPa during retention at 950°C. After retention ended, it was kept in the furnace to allow natural cooling.

30 [0121] The sintered compact was ground with a surface grinder to give a disk-shaped target with a diameter of 164 mm and a thickness of 3 mm. A Co-Pt-Cr-Ta₂O₅-SiO₂ based target having fine oxide grains with an average grain

diameter of 10 nm.

Subsequently, the target was subjected to bonding on a Cu backing plate having a diameter of 180 mm and a thickness of 4 mm, and then placed in DC magnetron sputtering equipment to perform sputtering. The sputtering conditions were a sputtering power of 1 kW and an Ar gas pressure of 1.5 MPa, and pre-sputtering was performed at 2 kW; and thereafter, deposition was performed on a Si substrate having a diameter of 4 inches at 1 kW for 200 seconds. The number of particles adhered on the substrate was measured with a particle counter, which was repeated 3 times in the same sputtering conditions.

The average number of particles on the Si substrate in this case was 0.3 having a particle size of 0.17 μm to 0.25 μm , and 0 having a particle size of 0.25 to 3.0 μm . The results are shown in Table 1.

A target having such fine grains did not generate abnormal electric discharge due to oxides during sputtering, but enabled to reduce particle generation. Accordingly, stable electric discharge was obtained when sputtering was performed with magnetron sputtering equipment.

According to the present invention, the structure of a ferromagnetic sputtering target, in particular the shape of oxide grains can be adjusted (micronized) to reduce contamination of impurities from a grinding device and a medium. Therefore, abnormal electric discharge due to oxides during sputtering will not occur, and particle generation can be reduced. Thus, when the target of the present invention is used, stable electric discharge can be obtained during sputtering with a magnetron sputtering device. Further, it is useful as a ferromagnetic sputtering target used for deposition of a magnetic thin film in a magnetic recording medium, in particular, of a hard disk drive recording layer for the following excellent effects: abnormal electric discharge due to oxides can be suppressed; particle generation during sputtering caused by abnormal electric discharge can be decreased; and a cost reducing effect by higher yields can be obtained.

CLAIMS

1. An oxide-containing magnetic material sputtering target, wherein the oxides have an average grain diameter of 400 nm or less.

2. The oxide-containing magnetic material sputtering target according to claim 1, comprising Phase A in which the oxides have an average grain diameter of 400 nm or less, and Phase B in which the oxides surrounding Phase A have an average grain diameter of 2 μ m or less.

3. The oxide-containing magnetic material sputtering target according to any one of claims 1 to 2, having an oxide structure in which an average grain diameter is 400 nm or less and an aspect ratio is 2 or less.

4. The oxide-containing magnetic material sputtering target according to any one of claims 1 to 3, comprising Phase A having an oxide structure in which an average grain diameter is 400 nm or less and the aspect ratio is 2 or less for a rectangular circumscribing an oxide grain to have an area thereof at the minimum, and Phase B in which the oxides surrounding Phase A have an average grain diameter of 2 μ m or less.

5. The magnetic material sputtering target according to any one of claims 1 to 4, wherein Cr is 45 mol% or less (except 0 mol%) and Co.

6. The magnetic material sputtering target according to any one of claims 1 to 4, wherein Cr is 45 mol% or less (except 0 mol%), Pt is 1 mol% or more and 30 mol% or less, and Co.

7. The magnetic material sputtering target according to any one of claims 1 to 4, wherein Pt is 1 mol% or more and 30 mol% or less and Co.

8. The magnetic material sputtering target according to any one of claims 1 to 4, wherein Pt is 5 mol% or more and 60 mol% or less and Fe.

9. The magnetic material sputtering target according to any one of claims 1 to 8, wherein as an additive element, one or more elements selected from B, Ti, V, Mn, Zr, Nb, Ru, Mo, Ta, W, Ag, Au, Cu and C in an amount of 0.5 mol% or more and 20 mol% or less is added.

10. The magnetic material sputtering target according to any one of claims 1 to 9, wherein as an oxide raw material, an oxide of one or more components selected from B, Si, Cr, Ti, Ta, W, Al, Mg, Mn, Ca, Zr, and Y in an amount of 1 to 20 mol% is added.

11. The magnetic material sputtering target according to any one of claims 1 to 10, wherein as an additive material, an inorganic substance material having one or more components selected from carbon, nitride and carbide is added.

12. The magnetic material sputtering target according to any one of claims 1 to 11, wherein HIP compaction treatment is performed after hot pressing.

13. A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, and further sintering the raw material.

14. The method of producing the oxide-containing magnetic material sputtering target according to any one of claims 1 to 12, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, and further sintering the raw material.

15. A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, further mixing the raw material with added insufficient components, and sintering the mixture.

16. The method of producing the oxide-containing magnetic material sputtering target according to any one of claims 1 to 12, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, pulverizing the material to obtain a material for the target, further mixing the raw material with added insufficient components, and sintering the mixture.

17. A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, and further performing hot isostatic pressing (HIP) on the resulting magnetic material.

18. The method of producing the oxide-containing magnetic material sputtering target according to any one of claims 1 to 12, by depositing a magnetic material on a substrate by PVD or CVD, removing the substrate from the deposited magnetic material, and further performing hot isotactic pressing (HIP) on the resulting magnetic material.

19. A method of producing an oxide-containing magnetic material sputtering target, by depositing a magnetic material on a substrate by PVD or CVD, and removing the substrate from the deposited magnetic material to obtain a target.

20. The method of producing the oxide-containing magnetic material sputtering target according to any one of claims 1 to 12, by depositing a magnetic material on a substrate by PVD or CVD, and removing the substrate

from the deposited magnetic material to obtain a target.

21. The method of producing the oxide-containing magnetic material sputtering target according to any one of claims 17 to 20, by removing the substrate from the deposited magnetic material, and laminating the obtained thin film to obtain a target.