

## DESCRIPTION

## SPUTTERING TARGET

## [Technical Field]

[0001] The present invention relates to a sputtering target to be used for forming magnetic thin films of magnetic recording media.

## BACKGROUND

[0002] In the field of magnetic recording as represented with hard disk drives, a material based on Co, Fe or Ni as ferromagnetic metals is being used as the material of the magnetic thin film in a magnetic recording medium. For example, Co-Cr-based or Co-Cr-Pt-based ferromagnetic alloy with Co as its main component is used for the magnetic thin film of hard disks adopting the longitudinal magnetic recording system.

Composite materials of Co-Cr-Pt based ferromagnetic alloys with Co as its main component and an oxide are often used for the magnetic thin film of hard disks adopting the vertical magnetic recording system which was recently put into practical application. In addition, the magnetic thin film is often produced by sputtering, via a magnetron sputtering device, a sputtering target having the foregoing materials as its components in light of its high productivity.

[0003] Incidentally, the recording density of a hard disk is rapidly increasing year by year and is in the process of exceeding 1 Tbit/in<sup>2</sup>. If the recording density reaches 1 Tbit/in<sup>2</sup>, the size of the recording bit will fall below 10 nm. In such a case, it is anticipated that the superparamagnetism caused by thermal fluctuation will become a problem, and it is further anticipated that the currently used magnetic recording medium material; for instance, a material with higher magnetic crystalline anisotropy obtained by adding Pt to a Co-Cr-based alloy, will no longer be sufficient. This is because, for particles to stably behave ferromagnetically at a size of 10 nm or less, the magnetic grains need to possess even higher magnetic crystalline anisotropy.

[0004] Based on the above-described circumstances, Fe-Pt alloys having an L1<sub>0</sub> structure has attracted attention as a material for ultra-high density recording media. The Fe-Pt alloys having a L1<sub>0</sub> structure have not only high magnetocrystalline anisotropy but also excellent corrosion resistance and

oxidation resistance and are therefore expected as a material that can be suitably applied to magnetic recording media.

[0005] Unfortunately, an Fe-Pt film formed by sputtering is a disordered phase, which is a metastable phase, and is therefore required to be converted into an L1<sub>0</sub> structure, which is an ordered phase, by thermal treatment at an ordering temperature. This ordering temperature is high and causes a problem of heat resistance of a substrate. Accordingly, a reduction in the ordering temperature by adding Ag or Cu to the Fe-Pt alloy has been tried.

[0006] Furthermore, in order to use an Fe-Pt alloy as a material for an ultra-high density recording medium, it is necessary to develop a technology of dispersing the Fe-Pt alloy in the form of magnetic grains having the L1<sub>0</sub> structure in a magnetically isolated state being aligned in the same orientation with a high density as much as possible.

On the basis above, a magnetic thin film having a granular structure, in which magnetic grains are isolated from one another by a nonmagnetic material such as an oxide or carbon, has been proposed for magnetic recording media of the next-generation hard disks. In this magnetic thin film having a granular structure, the magnetic grains are magnetically insulated from one another by means of the intervention of the nonmagnetic material. For example, Patent Literature 1 describes a magnetic recording medium including a magnetic thin film having a granular structure.

[0007] Such a magnetic thin film having a granular structure is conventionally produced by co-sputtering targets of individual materials in many cases. However, since co-sputtering apparatuses are expensive and large-sized, a monolithic sputtering target composed of an Fe-Pt based alloy and a nonmagnetic material is generally used in mass production of magnetic thin films. Such a target is produced by the powder sintering method.

[0008] However, when a sputtering target made from an Fe-Pt alloy and C is sputtered, there is a problem in that C becomes inadvertently detached from the sputtering surface during sputtering and become adhered to the substrate. This substance generated is called particles. The floating amount of a magnetic head is reduced in accordance with an increase in the recording density of a hard disk drive. Thus, the acceptable size and number of particles on a magnetic recording medium have become increasingly restricted.

Patent documents relating to sputtering targets for recording media including Fe-Pt based materials are shown below for reference.

[Prior Art Document]

[Patent Document]

[0009] Patent Literature 1: Japanese Patent Laid-Open No. 2004-152471  
Patent Literature 2: Japanese Patent Laid-Open No. 2003-313659  
Patent Literature 3: Japanese Patent Laid-Open No. 2011-210291  
Patent Literature 4: Japanese Patent No. 5041262  
5 Patent Literature 5: Japanese Patent No. 5041261  
Patent Literature 6: Japanese Patent Laid-Open No. 2012-214874

## SUMMARY OF INVENTION

### [Technical Problem]

10 [0010] It is an object of the present invention to provide a sputtering target comprising an Fe-Pt based alloy, silver (Ag), and C, in which the amount of particles generated during sputtering is significantly reduced.

### [Solution to Problem]

15 [0011] As a result of intensive study to solve the above problem, the present inventors have discovered that the amount of particles generated during sputtering can be significantly reduced by adjusting the composition and the microstructure of the sputtering target. Here in the present invention, analysis of the microstructure of a target is performed for a cross section perpendicular to the sputtering surface of the target. A target section or cross-section described hereinafter refers to a section perpendicular to the sputtering surface.

20 [0012] Based on the foregoing discovery, the present invention provides:

- 25 1) A sputtering target comprising an Fe-Pt based alloy phase, a Ag phase, and a C phase, wherein a value obtained by dividing the atomic number ratio of C by the atomic number ratio of Ag is 4 to 10 in the composition of the entire sputtering target; and the Ag phase has a shape that is included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape that has at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase, in a cross section of the sputtering target;
- 30 2) The sputtering target according to 1) above, wherein the C phase has an area ratio of 10% to 45% in a cross section of the sputtering target;
- 35 3) A sputtering target comprising an Fe-Pt based alloy phase, a Ag phase, a C phase, and an oxide phase, wherein a value obtained by dividing the atomic number ratio of C by the atomic number ratio of Ag is 4 to 10 in the composition of the entire sputtering target; and the Ag phase has a shape that is included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape that has at least two tangent points or

intersection points between the imaginary circle and the periphery of the Ag phase, in a cross section of the sputtering target;

4) The sputtering target according to 3) above, wherein the oxide phase comprises an oxide including a component that is at least one element selected from the group consisting of Al, B, Ba, Be, Bi, Ca, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Ho, La, Li, Lu, Mg, Mo, Nb, Nd, Ni, Pr, Sb, Sc, Si, Sm, Sn, Sr, Ta, Tb, Te, Ti, Tm, V, W, Y, Yb, Zn, and Zr;

5) The sputtering target according to 3) or 4) above, wherein a sum of the area ratio of the C phase and the oxide phase is 10% to 45% in a cross section of the sputtering target; and

6) The sputtering target according to any one of 1) to 5) above, comprising at least one element selected from the group consisting of Au, B, Co, Cr, Cu, Ga, Ge, Mn, Mo, Nb, Ni, Pd, Re, Rh, Ru, Sn, Ta, W, V, and Zn in an amount such that the atomic number ratio of the element(s) is 0.5% to 15% of the total metal components.

#### [Effects of Invention]

[0013] The present invention is able to provide a sputtering target that can significantly reduce the amount of particles generated during sputtering. The sputtering target accordingly has an effect of increasing the yield in sputtering film formation.

#### BRIEF DESCRIPTION OF DRAWINGS

[0014]

Fig. 1 includes schematic diagrams illustrating examples of the shape of Ag phases of the present invention.

Fig. 2 is a schematic diagram illustrating an example of the shape of a Ag phase that is outside the scope of the present invention.

Fig. 3 includes two-dimensional images and elemental distribution images of cross sections of the sputtering target in Example 1 observed with an electron beam probe microanalyzer (EPMA).

#### DETAILED DESCRIPTION

[0015] The sputtering target of the present invention is characterized by comprising an Fe-Pt based alloy phase, a Ag phase, and a C phase. Herein, the Fe-Pt based alloy phase refers to an alloy comprising Fe and Pt as main components, by which refers not only a binary system alloy containing only Fe

and Pt but also a ternary or higher system alloy containing a metal element or elements in addition to Fe and Pt, such as Fe-Pt-Cu, for example.

[0016] The sputtering target of the present invention is characterized in that the value obtained by dividing the atomic number ratio of C by the atomic number ratio of Ag is 4 to 10 in the composition of the entire sputtering target. The Ag phase has an effect of inhibiting detachment of C from the target surface during sputtering and thereby can significantly inhibit the generation of particles. Studies by the present inventors have revealed that the effect of inhibiting the generation of particles is significantly reduced if the value obtained by dividing the atomic number ratio of C by the atomic number ratio of Ag is less than 4. In addition, if this value is larger than 10, although the effect of inhibiting the generation of particles is maintained, Ag segregates in the sputtered film in some cases, and the magnetic characteristics of the magnetic thin film may deteriorate.

[0017] The sputtering target of the present invention is characterized in that the Ag phase has a shape that is included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape that has at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase, in a cross section of the target. This characteristic means that the target structure substantially does not contain coarse Ag phases. If a target structure contains coarse Ag phases, Ag having a high sputtering rate is selectively sputtered to cause problems of deteriorating the smoothness of the sputtering surface and increasing the amount of particles. Even in the case of a sputtering target including coarse Ag phases not having the above-described shapes, however, the advantageous effect of the present invention can be achieved provided that the area ratio of the coarse Ag phases is less than 20% of the total area of the Ag phases.

[0018] In the sputtering target of the present invention, the area ratio of the C phase in a cross section of the target is preferably 10% to 45%. If the area ratio of the C phase is less than 10%, C in the sputtering film cannot sufficiently insulate the magnetic interaction between magnetic grains, and satisfactory magnetic characteristics may not be obtained. An area ratio of higher than 45% may cause aggregation of C to generate coarse C phases in the target structure and to increase the generation of particles. The area ratio of the C phase is preferably determined from the average of the area ratios that are obtained through observation of a plurality of cross-sections of about 1  $\text{mm}^2$  in total for reducing the variation due to the observation position.

[0019] The sputtering target of the present invention can further contain an oxide phase in the structure comprising the Fe-Pt based alloy phase, the Ag phase,

and the C phase. The oxide phase is effective for insulating the magnetic interaction between the magnetic grains in the sputtered film as in C.

Examples of the oxide phase include those each composed of an oxide including a component that is at least one element selected from the group consisting of Al, B, Ba, Be, Bi, Ca, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Ho, La, Li, Lu, Mg, Mo, Nb, Nd, Ni, Pr, Sb, Sc, Si, Sm, Sn, Sr, Ta, Tb, Te, Ti, Tm, V, W, Y, Yb, Zn, and Zr. These oxides can be arbitrary selected depending on the desired magnetic characteristics.

[0020] In the sputtering target containing the oxide phase of the present invention, preferably, the total area ratio of the C phase and the oxide phase is 10% to 45%. If the total area ratio of the C phase and the oxide phase is less than 10%, C and the oxide in the sputtering film cannot sufficiently insulate the magnetic interaction between magnetic grains, and satisfactory magnetic characteristics may not be obtained. If a total area ratio is higher than 45%, aggregation of C and the oxide may cause to generate coarse C phases and oxide phases in the target and to increase the generation of particles. Incidentally, the area ratio of the C phase and the oxide phase is preferably determined from the averages of the area ratio that are obtained through observation of a plurality of cross-sections of approximately 1 mm<sup>2</sup> in total in order to reduce the variation due to the observation position.

[0021] The sputtering target of the present invention preferably contain at least one metal element selected from the group consisting of Au, B, Co, Cr, Cu, Ga, Ge, Mn, Mo, Nb, Ni, Pd, Re, Rh, Ru, Sn, Ta, W, V, and Zn. The atomic number ratio of the metal element in the metal components preferably is 0.5% to 15%. The additional metals are added mainly to reduce the temperature of the heat treatment for obtaining the L1<sub>0</sub> structure. A content of less than 0.5% can scarcely provide the above-mentioned effect, whereas a content of greater than 15% may deteriorate the magnetic characteristics of the magnetic thin film.

[0022] The sputtering target of the present invention may be produced via the powder sintering method, for example, and by the following process.

Metal powders of Fe, Pt, and Ag and an additional metal powder, as needed, are prepared. The powders may be metal powders of single elements or may be alloy powders. Preferably, each of these metal powders has an average grain size of 1 to 10 μm. An average grain size of 1 to 10 μm can provide a highly uniform mixture and can be prevented from segregating and coarsening in the target structure. If the average grain size of the metal powder is larger than 10 μm, the C phase and the oxide phase may not be uniformly dispersed, whereas an average grain size of smaller than 1 μm may cause a

problem of influence due to oxidation of the metal powder. These ranges of the grain size are, however, merely preferred conditions, and grain sizes outside these ranges are also within the scope of the present invention. A Ag powder readily aggregates depending on the grain size or the shape. Such a Ag powder is preferably subjected to coating to prevent aggregation.

[0023] The C powder preferably has an average grain size of 1 to 30  $\mu\text{m}$ . An average grain size of 1 to 30  $\mu\text{m}$  can prevent the C powder mixed with a metal powder from aggregating and can be uniformly dispersed. However, this range of the grain size is a preferred condition, and grain sizes outside this range are also within the scope of the present invention. The type of the C phase may have a crystal structure, such as graphite and nanotubes, or may be amorphous, such as carbon black.

[0024] The oxide powder preferably has an average grain size of 0.2 to 5  $\mu\text{m}$ . An average grain size of 0.2 to 5  $\mu\text{m}$  has an advantage of being readily and uniformly mixed with metal powders. If the average grain size of the oxide powder is larger than 5  $\mu\text{m}$ , coarse oxide phases may occur after sintering, whereas an average grain size of less than 0.2  $\mu\text{m}$  may cause aggregation of the oxide powder. However, this range of the grain size is merely a preferred condition, and grain sizes outside this range are also within the scope of the present invention.

[0025] The above-described raw material powders are weighed to give a desired composition and are mixed and pulverized through a known process with, for example, a ball mill. On this occasion, an inert gas is preferably sealed in the pulverizing vessel for inhibiting oxidation of the raw material powders.

[0026] The thus-prepared powder mixture is molded and sintered via the hot-pressing method in a vacuum atmosphere or in an inert gas atmosphere. The molding and sintering may be performed by various pressurized sintering, such as spark plasma sintering, instead of the hot-pressing method. In particular, hot hydrostatic pressure sintering is effective for increasing the density of the sintered compact.

Although the holding temperature during sintering varies depending on the components of the target, the holding temperature is preferably within a range of 500°C to 950°C in many cases. Further, the holding temperature is preferably 900°C or less so that in a cross-section of the target, a Ag phase can have a shape that is smaller than every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape that has at least two tangent points or intersection points between the imaginary circle and the

periphery of the Ag phase. When the holding temperature is higher than 900°C, the Ag grain will grow and make it hard to satisfy the above characteristics.

[0027] The obtained sintered compact is processed into a desired target shape with, for example, a lathe to produce the sputtering target of the present invention.

5 The produced sputtering target can reduce the amount of particles generated during sputtering and thereby has an advantageous effect of increasing the yield in sputtering film formation.

### Examples

10 [0028] The present invention will now be described by examples and comparative examples. These examples are merely exemplary and are not intended to limit the scope of the present invention. Namely, the present invention is limited only by the claims and encompasses various modifications in addition to examples included in the present invention.

15 [0029]

#### (Example 1)

20 An Fe powder having an average grain size of 3  $\mu\text{m}$ , a Pt powder having an average grain size of 3  $\mu\text{m}$ , a Ag powder having an average grain size of 2  $\mu\text{m}$ , and a C powder (graphite powder) having an average grain size of 10  $\mu\text{m}$  were prepared as raw material powders. An Ag powder subjected to coating treatment with an organic material was used to prevent aggregation.

The powders were weighed to attain a total weight of 2500 g based on the following composition ratio:

Weighed composition (molar fraction): 30Fe-30Pt-5Ag-35C.

25 [0030] Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

35 [0031] The obtained sintered compact was partially cut out, and the cross section was polished and observation with an electron beam probe microanalyzer (EPMA) was performed thereto. As a result, a microstructure including mutually uniformly dispersed Fe-Pt alloy phase, Ag phase, and C phase was detected. In

addition, an area of 0.2 mm<sup>2</sup> of the cross section was observed with an EPMA, and it was detected that in 80% or more of the Ag phase in the area ratio, the Ag phase had a shape being included in every imaginary circle having a radius of 10 μm drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. Further, when the area ratio of the C phase was determined from an image of an area of 1 mm<sup>2</sup> taken with an optical microscope, it was 25.5%.

[0032] Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, and Ag were measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 6.9.

[0033] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering.

The sputtering conditions were input power of 1 kW and Ar gas pressure of 1.7 Pa; and after performing pre-sputtering at 2 kW/hr, a film was deposited for 20 seconds on a silicon substrate having a 4-inch diameter. Subsequently, the number of particles adhered onto the substrate was counted with a particle counter. The number of particles was 83.

[0034] (Comparative Example 1)

An Fe powder having an average grain size of 3 μm, a Pt powder having an average grain size of 3 μm, a Ag powder having an average grain size of 2 μm, and a C powder (graphite powder) having an average grain size of 10 μm were prepared as raw material powders. An Ag powder subjected to coating treatment with an organic material was used to prevent aggregation.

The powders were weighed to attain a total weight of 2500 g based on the following composition ratio:

Weighed composition (molar fraction): 30Fe-30Pt-2Ag-38C.

[0035] Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and

molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

[0036] The obtained sintered compact was partially cut out, and the cross section was polished and observation with an electron beam probe microanalyzer (EPMA) was performed thereto. As a result, a microstructure including mutually uniformly dispersed Fe-Pt alloy phase, Ag phase, and C phase was detected. In addition, an area of 0.2 mm<sup>2</sup> of the cross section was observed with an EPMA, and it was detected that in 80% or more of the Ag phase in the area ratio, the Ag phase had a shape being included in every imaginary circle having a radius of 10 μm drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. Further, when the area ratio of the C phase was determined from an image of an area of 1 mm<sup>2</sup> taken with an optical microscope, it was 28.4%.

[0037] Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, and Ag were measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 18.5. The value was outside the range defined by the claims of the present application.

[0038] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as in Example 1. The number of particles was 561, and the number increased significantly as compared with Example 1.

[0039]

#### (Comparative Example 2)

An Fe powder having an average grain size of 3 μm, a Pt powder having an average grain size of 3 μm, a Ag powder having an average grain size of 30 μm, and a C powder (graphite powder) having an average grain size of 10 μm

were prepared as raw material powders. The powders were weighed to attain a total weight of 2500 g based on the following composition ratio:

Weighed composition (molar fraction): 30Fe-30Pt-5Ag-35C.

[0040]

Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 960°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

[0041]

The obtained sintered compact was partially cut out, and the cross section was polished and observation with an electron beam probe microanalyzer was performed thereto. As a result, a microstructure including mutually uniformly dispersed Fe-Pt alloy phase, Ag phase, and C phase was detected. In addition, observation of an area of 0.2 mm<sup>2</sup> of the cross section was performed and it was detected that coarse Ag phases were present and that in 20% or more of the total area of the Ag phase in the area ratio, the Ag phase did not have a shape being included in every imaginary circle having a radius of 10 μm drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. This occurred probably because the Ag powder having a large grain size was used and sintering was performed at a high temperature. Further, when the area ratio of the C phase was determined from an image of an area of 1 mm<sup>2</sup> taken with an optical microscope, it was 25.5%.

[0042]

Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, and Ag were measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 7.0.

35 [0043]

The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as

in Example 1. The number of particles was 245, which was larger in number as compared with Example 1.

[0044]

(Comparative Example 3)

5 An Fe powder having an average grain size of 3  $\mu\text{m}$ , a Pt powder having an average grain size of 3  $\mu\text{m}$ , a Ag powder having an average grain size of 2  $\mu\text{m}$ , and a C powder (graphite powder) having an average grain size of 10  $\mu\text{m}$  were prepared as raw material powders. An Ag powder subjected to coating treatment with an organic material was used to prevent aggregation.

10 The powders were weighed to attain a total weight of 1800 g based on the following composition ratio:

Weighed composition (molar fraction): 17Fe-17Pt-6Ag-60C.

[0045]

15 Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

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[0046]

25 A cross section of the obtained sintered compact was polished, and observation of the cross section was performed thereto with an electron beam probe microanalyzer (EPMA). As a result, a microstructure including mutually uniformly dispersed Fe-Pt alloy phase, Ag phase, and C phase was detected. In addition, an area of 0.2  $\text{mm}^2$  of the cross section was observed with an EPMA, and it was detected that in 80% or more of the Ag phase in the area ratio, the Ag phase had a shape being included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. Further, when the area ratio of the C phase was determined from an image of an area of 1  $\text{mm}^2$  taken with an optical microscope, it was 48.1%, which was outside the range defined by the claims of the present application.

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[0047]

Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, and Ag were measured using an ICP-AES device, and C was measured using an oxygen analyzer

adopting inert gas fusion-infrared absorption. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 9.8.

[0048] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as in Example 1. The number of particles was 880, and the number increased significantly as compared with Example 1.

(Example 2)

An Fe powder having an average grain size of 3  $\mu\text{m}$ , a Pt powder having an average grain size of 3  $\mu\text{m}$ , a Ag powder having an average grain size of 2  $\mu\text{m}$ , a C powder (graphite powder) having an average grain size of 10  $\mu\text{m}$ , and a  $\text{SiO}_2$  powder having an average grain size of 1  $\mu\text{m}$  were prepared as raw material powders. An Ag powder subjected to coating treatment with an organic material was used to prevent aggregation.

The powders were weighed to attain a total weight of 2200 g based on the following composition ratio:

Weighed composition (molar fraction): 30Fe-30Pt-5Ag-30C-5 $\text{SiO}_2$ .

[0050] Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

[0051] The obtained sintered compact was partially cut out, and the cross section was polished and observation with an electron beam probe microanalyzer (EPMA) was performed thereto. As a result, a microstructure including mutually uniformly dispersed Fe-Pt alloy phase, Ag phase, C phase, and oxide ( $\text{SiO}_2$ ) phase were detected. In addition, an area of 0.2  $\text{mm}^2$  of the cross section was observed with an EPMA, and it was detected that in 80% or more of the Ag phase in the area ratio, the Ag phase had a shape being included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary

point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. Further, when the total area ratio of the C phase and the oxide phase was determined from an image of an area of 1 mm<sup>2</sup> taken with an optical microscope, it was 35.5%.

[0052] Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, Ag, and Si were measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace; and O was measured using an oxygen analyzer adopting inert gas fusion-infrared absorption. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 6.0.

[0053] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as in Example 1. The number of particles was 34.

[0054]

#### (Comparative Example 4)

An Fe powder having an average grain size of 3 μm, a Pt powder having an average grain size of 3 μm, a Ag powder having an average grain size of 2 μm, a C powder (graphite powder) having an average grain size of 10 μm, and a SiO<sub>2</sub> powder having an average grain size of 1 μm were prepared as raw material powders. An Ag powder subjected to coating treatment with an organic material was used to prevent aggregation.

The powders were weighed to attain a total weight of 2300 g based on the following composition ratio:

Weighed composition (molar fraction): 32.5Fe-32.5Pt-2Ag-28C-5SiO<sub>2</sub>.

[0055]

Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, of a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature

until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

[0056] A cross section of the obtained sintered compact was polished, and observation of the cross section was performed thereto with an electron beam probe microanalyzer (EPMA). As a result, a microstructure including mutually uniformly dispersed Fe-Pt alloy phase, Ag phase, C phase, and oxide ( $\text{SiO}_2$ ) phase was detected. In addition, an area of  $0.2 \text{ mm}^2$  of the cross section was observed with an EPMA, and it was detected that in 80% or more of the Ag phase in the area ratio, the Ag phase had a shape being included in every imaginary circle having a radius of  $10 \text{ }\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. Further, the total area ratio of the C phase and the oxide phase was determined from an image of an area of  $1 \text{ mm}^2$  taken with an optical microscope, it was 34.2%.

[0057] Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, Ag, and Si were measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace; and O was measured using an oxygen analyzer adopting inert gas fusion-infrared absorption. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 13.9. The value was outside the range defined by the claims of the present application.

[0058] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as in Example 2. The number of particles was 189, and the number increased significantly as compared with Example 2.

[0059] (Comparative Example 5)

An Fe powder having an average grain size of  $3 \text{ }\mu\text{m}$ , a Pt powder having an average grain size of  $3 \text{ }\mu\text{m}$ , a Ag powder having an average grain size of  $2 \text{ }\mu\text{m}$ , and a C powder (graphite powder) having an average grain size of  $10 \text{ }\mu\text{m}$  were prepared as raw material powders. An Ag powder subjected to coating treatment with an organic material was used in order to prevent aggregation.

The powders were weighed to attain a total weight of 1700 g based on the following composition ratio:

Weighed composition (molar fraction): 24Fe-24Pt-5Ag-40C-7SiO<sub>2</sub>.

[0060] Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

[0061] A cross section of the thus obtained sintered compact was polished, and observation of the cross section was performed thereto with an electron beam probe microanalyzer (EPMA). As a result, a microstructure including mutually uniformly dispersed Fe-Pt alloy phase, Ag phase, C phase, and oxide (SiO<sub>2</sub>) phase was detected. In addition, an area of 0.2 mm<sup>2</sup> of the cross section was observed with an EPMA, and it was detected that in 80% or more of the Ag phase in the area ratio, the Ag phase had a shape being included in every imaginary circle having a radius of 10 μm drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. Further, when the total area ratio of the C phase and the oxide phase was determined from an image of an area of 1 mm<sup>2</sup> taken with an optical microscope, it was 47.4%, which was outside the range defined by the claims of the present application.

[0062] Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, Ag, and Si were measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace and O was measured using an oxygen analyzer adopting inert gas fusion-infrared absorption. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 7.9.

[0063] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as

in Example 1. The number of particles was 716, and the number increased significantly as compared with Example 1.

[0064]

(Example 3)

5           An Fe powder having an average grain size of 3  $\mu\text{m}$ , a Pt powder having an average grain size of 3  $\mu\text{m}$ , a Ag powder having an average grain size of 2  $\mu\text{m}$ , a Cu powder having an average grain size of 3  $\mu\text{m}$ , a Co powder having an average grain size of 3  $\mu\text{m}$ , and a C powder (graphite powder) having an average grain size of 10  $\mu\text{m}$  were prepared as raw material powders. An Ag powder subjected to coating treatment with an organic material was used to prevent aggregation.

The powders were weighed to attain a total weight of 2200 g based on the following composition ratio:

Weighed composition (molar fraction): 26Fe-26Pt-6Cu-5Co-7Ag-30C.

15 [0065]

Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

25 [0066]

The obtained sintered compact was partially cut out, and the cross section was polished and observation with an electron beam probe microanalyzer (EPMA) was performed thereto. As a result, a microstructure including mutually uniformly dispersed Fe-Pt-Cu-Co alloy phase, Ag phase, and C phase were detected. In addition, an area of 0.2 mm<sup>2</sup> of the cross section was observed, and it was detected that in 80% or more of the Ag phase in the area ratio, the Ag phase had a shape being included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. Further, when the area ratio of the C phase was determined from an image of an area of 1 mm<sup>2</sup> taken with an optical microscope, it was 21.6%.

30

35

[0067]

Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, Ag, Cu, and Co were

measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the

5 [0068] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as

10 [0069] (Comparative Example 6)

An Fe powder having an average grain size of 3  $\mu\text{m}$ , a Pt powder having an average grain size of 3  $\mu\text{m}$ , a Ag powder having an average grain size of 2  $\mu\text{m}$ ,  
15 a Cu powder having an average grain size of 3  $\mu\text{m}$ , a Co powder having an average grain size of 3  $\mu\text{m}$ , and a C powder (graphite powder) having an average grain size of 10  $\mu\text{m}$  were prepared as raw material powders. An Ag powder which was not subjected to coating treatment with an organic material was used to prevent aggregation.

20 The powders were weighed to attain a total weight of 2200 g based on the following composition ratio:

Weighed composition (molar fraction): 26Fe-26Pt-6Cu-5Co-7Ag-30C.

[0070] Subsequently, the weighed powders were put in a ball mill pot having a capacity of 10 liters together with zirconia balls as the pulverizing medium, sealed  
25 in an Ar atmosphere, and mixed and pulverized by rotating the pot for 4 hours. The powder removed from the pot was thereafter filled in a carbon mold and molded and sintered using a hot-pressing machine. The hot-pressing conditions were as follows; namely, a vacuum atmosphere, a rate of temperature increase of 300°C/hour, a holding temperature of 900°C, a holding time of 2 hours, and a  
30 pressure of 30 MPa from the start of the increase in temperature until the end of holding. After the end of holding, the resultant product was naturally cooled as is in the chamber.

[0071] The obtained sintered compact was partially cut out, and the cross section was polished and observation with an electron beam probe microanalyzer  
35 (EPMA) was performed thereto. As a result, a microstructure including mutually uniformly dispersed Fe-Pt-Cu-Co alloy phase, Ag phase, and C phase were detected. In addition, an area of 0.2 mm<sup>2</sup> of the cross section was observed with an EPMA, and it was detected that coarse Ag phases were present and that

in 20% or more of the Ag phase in the area ratio, the Ag phase did not have a shape being included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase. This occurred probably because the Ag powder without treatment to prevent aggregation was used. Further, when the area ratio of the C phase was determined from an image of an area of 1  $\text{mm}^2$  taken with an optical microscope, it was 21.7%.

[0072] Subsequently, a small piece extracted from the produced sintered compact was subjected to composition analysis. Fe, Pt, and Ag were measured using an ICP-AES device, and C was measured using a carbon analyzer adopting an infrared absorption method after combustion in a high-frequency induction furnace. As a result of calculating the atomic number ratio from the obtained weight ratios, the atomic number ratio of C divided by the atomic number ratio of Ag was 4.2.

[0073] The sintered compact was then cut with a lathe to obtain a disk-shaped target having a diameter of 180.0 mm and a thickness of 5.0 mm. The target was placed in a magnetron sputtering apparatus (C-3010 system, manufactured by Canon ANELVA Corporation) to perform sputtering in the same conditions as in Example 1. The number of particles was 183, which was larger in number as compared with Example 3.

[0074] As described above, it was found out that in every example, the amount of particles generated during sputtering can be reduced and that the reduction is very important for increasing the yield in film formation.

It should be noted that although targets containing Cu and Co as additional metals have been exemplarily described in the examples, the same results were obtained in cases when targets contain these metal elements: Au, B, Cr, Ga, Ge, Mn, Mo, Nb, Ni, Pd, Re, Rh, Ru, Sn, Ta, W, V, and Zn.

[0075] [Table 1]

|                       | Weighted composition (molar fraction)   | C/Ag atomic number ratio | Volume ratio of C phase (%) | Sum of volume ratio of C phase and oxide phase (%) | Shape of Ag phase       | Number of particles |
|-----------------------|-----------------------------------------|--------------------------|-----------------------------|----------------------------------------------------|-------------------------|---------------------|
| Example 1             | 30Fe-30Pt-5Ag-35C                       | 6.9                      | 25.5                        | —                                                  | within specified range  | 83                  |
| Comparative Example 1 | 30Fe-30Pt-2Ag-38C                       | 18.5                     | 28.4                        | —                                                  | within specified range  | 561                 |
| Comparative Example 2 | 30Fe-30Pt-5Ag-35C                       | 7.0                      | 25.5                        | —                                                  | outside specified range | 245                 |
| Comparative Example 3 | 17Fe-17Pt-6Ag-60C                       | 9.8                      | 48.1                        | —                                                  | within specified range  | 880                 |
| Example 2             | 30Fe-30Pt-5Ag-30C-5SiO <sub>2</sub>     | 6.0                      | —                           | 35.5                                               | within specified range  | 34                  |
| Comparative Example 4 | 32.5Fe-32.5Pt-2Ag-28C-5SiO <sub>2</sub> | 13.9                     | —                           | 34.2                                               | within specified range  | 189                 |
| Comparative Example 5 | 24Fe-24Pt-5Ag-40C-7SiO <sub>2</sub>     | 7.9                      | —                           | 47.4                                               | within specified range  | 716                 |
| Example 3             | 26Fe-26Pt-6Cu-5Co-7Ag-30C               | 4.2                      | 21.6                        | —                                                  | within specified range  | 17                  |
| Comparative Example 6 | 26Fe-26Pt-6Cu-5Co-7Ag-30C               | 4.2                      | 21.7                        | —                                                  | outside specified range | 183                 |

## [Industrial Applicability]

- 5 [0076] The sputtering target of the present invention has advantageous effects of significantly reducing the amount of particles generated during sputtering and increasing the yield in film formation. Accordingly, the sputtering target is useful for forming a magnetic thin film having a granular structure.

## CLAIMS

1. A sputtering target comprising an Fe-Pt based alloy phase, a Ag phase and a C phase, wherein a value obtained by dividing the atomic number ratio of C by the atomic number ratio of Ag is 4 or more and 10 or less in the composition of the entire sputtering target; the Ag phase has a shape being included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase, in a cross section of the sputtering target; and the C phase has an area ratio of 10% or more and 45% or less in a cross section of the sputtering target.

2. A sputtering target comprising a Ag phase, a C phase, and an oxide phase, wherein a value obtained by dividing the atomic number ratio of C by the atomic number ratio of Ag is 4 or more and 10 or less in the composition of the entire sputtering target, the Ag phase has a shape being included in every imaginary circle having a radius of 10  $\mu\text{m}$  drawn with its center at an arbitrary point in the Ag phase or a shape having at least two tangent points or intersection points between the imaginary circle and the periphery of the Ag phase, in a cross section of the sputtering target; and a sum of the area ratios of the C phase and the oxide phase is 10% or more and 45% or less in a cross section of the sputtering target.

3. The sputtering target according to Claim 2, wherein the oxide phase comprises an oxide including a component that is at least one element selected from the group consisting of Al, B, Ba, Be, Bi, Ca, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Ho, La, Li, Lu, Mg, Mo, Nb, Nd, Ni, Pr, Sb, Sc, Si, Sm, Sn, Sr, Ta, Tb, Te, Ti, Tm, V, W, Y, Yb, Zn, and Zr.

4.

The sputtering target according to any one of Claims 1, 2, and 3, comprising at least one element selected from the group consisting of Au, B, Co, Cr, Cu, Ga, Ge, Mn, Mo, Nb, Ni, Pd, Re, Rh, Ru, Sn, Ta, W, V, and Zn in an amount such that the atomic number ratio of the element(s) is 0.5% or more and 15% or less of the total metal components.