DESCRIPTION

Fe-Pt-BN-BASED SPUTTERING TARGET AND METHOD FOR MANUFACTURING SAME

TECHNICAL FIELD

[0001] The present invention relates to a BN-containing sputtering target to be used for producing a magnetic thin film as well as a production method therefor and particularly relates to an Fe-Pt-BN-based sputtering target containing Fe, Pt, and BN (boron nitride) as well as a production method therefor.

BACKGROUND ART

[0002] As a sputtering target for producing a granular magnetic thin film of a magnetic recording medium in a hard disk drive or the like, there has been used a sintered compact containing a ferromagnetic metal of Fe or Co as a main component and a nonmagnetic material, such as SiO_2 or another oxide, B (boron), C (carbon), or BN (boron nitride). Although exhibiting excellent performance as a lubricant, BN has problems of having difficulty in producing a high-density sintered compact due to inferior sinterability, of generating particles during sputtering and thereby lowering the product yield, of exhibiting poor machinability, and so forth.

[0003] To resolve these problems, there have been proposed, for example, a method of improving sinterability through alloying of BN with SiO_2 (Japanese Patent No. 5567227), a method of lowering the oxygen content of a sputtering target by using Fe-Pt alloy powder to suppress formation of iron oxide (Japanese Patent No. 5689543), and a method of improving sinterability through addition of AgCu alloy or AuCu alloy (Japanese Patent No. 6285043, Japanese Patent No. 6084711).

[0004] Japanese Patent No. 5567227 discloses that a high-density sputtering target which reduces particles to be generated during sputtering is provided by dispersing hexagonal BN grains as a nonmagnetic material, together with quartz or amorphous SiO_2 grains, in an Fe-Pt based parent metal and discloses that the sinterability of hexagonal BN can be enhanced

significantly by incorporating BN and, relative to BN, 1 mol% or more of SiO_2 in the state of solid solution. As a concrete production method, it is disclosed that raw material powders of Fe, Pt, SiO_2, and BN are mixed using a stirred media mill at 300 rpm for 2 hours and the resulting mixed powder is hot pressed and then subjected to hot isostatic pressing. Moreover, it is disclosed that the resulting sintered compact of Fe-Pt based magnetic material has, on the cross-section of the pressed surface, an X-ray diffraction peak intensity ratio of the (002) plane of hexagonal BN relative to the background intensity of 1.50 or more and an X-ray diffraction peak intensity ratio of the (101) plane of cristobalite, which is crystallized SiO_2, of 1.40 or less. Further, it is disclosed that the Comparative Examples (Fe-Pt-BN based, Fe-Pt-BN-oxide based, Fe-Pt-BN-nonmagnetic material based), which were produced under the same conditions but in the absence of SiO_2, exhibit an X-ray diffraction peak intensity ratio of the (002) plane of hexagonal BN comparable to the Examples but have a considerably increased number of particles as many as 645 or more. Meanwhile, since FE-EPMA observation results are not mentioned at all for the Comparative Examples, the dispersion states are unknown.

[0005] Japanese Patent No. 5689543 discloses that a sintered compact of Fe-Pt-BN based magnetic material having an oxygen content as low as 4,000 wtppm or less can be prepared by using Fe-Pt alloy powder and discloses that the prepared sintered compact exhibits satisfactory machinability and thus can suppress the occurrence of cracking or chipping. As a concrete production method, it is disclosed that BN powder and Fe-Pt alloy powder having a particle size of 0.5 μm or more and 10 μm or less are placed in a mortar and mixed uniformly and the resulting mixed powder is hot pressed and then subjected to hot isostatic pressing. Meanwhile, it is also disclosed that the Comparative Examples (Fe-Pt-BN based, Fe-Pt-BN-nonmagnetic material based), which were produced under the same conditions except for mixing Fe powder, Pt powder, and BN powder using a stirred media mill at 300 rpm for 2 hours, exhibited an oxygen content as high as 11,500 wtppm or more and that the formation of iron oxide and the occurrence of chipping were confirmed in these examples. Herein, it is explained that abnormal discharge or particle generation is suppressed when the

occurrence of chipping can be suppressed. However, since nothing is mentioned about particle generation in the Examples or the Comparative Examples, it is unknown whether particle generation could be suppressed.

[0006] Japanese Patent No. 6285043 and Japanese Patent No. 6084711 disclose, concerning an FePt-based sintered sputtering target containing C and/or BN, that the density of a sintered compact is increased at a low sintering temperature by adding a low-melting AgCu alloy or AuCu alloy. It is disclosed in Japanese Patent No. 6285043 that AgCu alloy consisting of Ag and Cu lowers its melting point to 779.1°C through the eutectic reaction and disclosed in Japanese Patent No. 6084711 that AuCu alloy consisting of Au and Cu lowers its melting point to 910°C through the eutectic reaction. Japanese Patent No. 6285043 discloses in Comparative Example 4 that Fe powder, Pt powder, BN powder, and Au powder were treated in a stirred media mill at 300 rpm for 2 hours, then mixed with Ag powder and BN powder in a V-type mixer, and further mixed using a 150 μm sieve and the resulting powder was sintered and subjected to hot isostatic pressing to exhibit a considerably increased number of particles as many as 833. Similarly, Japanese Patent No. 6084711 discloses that Fe powder, Pt powder, BN powder, and Au powder were treated in a stirred media mill at 300 rpm for 2 hours, then mixed with Au powder and BN powder in a V-type mixer, and further mixed using a 150 μm sieve and the resulting powder was sintered and subjected to hot isostatic pressing to exhibit a considerably increased number of particles as many as 256.

[0007] As in the foregoing, the above-mentioned patent literature discloses the techniques of suppressing particle generation in an Fe-Pt-BN based sintered compact by adding SiO_2, AgCu alloy, or AuCu alloy at the same level as main components or by lowering the oxygen content to 4,000 wtppm or less. However, when even one of these conditions is not satisfied, it has been impossible to reduce the number of particles. Moreover, as for the addition of SiO_2, AgCu alloy, or AuCu alloy at the same level as main components, when the desirable composition of a deposition film does not contain these elements, it may be impossible to attain desirable magnetic characteristics of a film due to a large difference from the desirable composition of a deposition film. Accordingly, when the desirable

composition of a deposition film does not contain SiO_2, AgCu alloy, or AuCu alloy, it is difficult to improve the sinterability of BN and thus suppress particle generation.

CITATION LIST

PATENT LITERATURE

[0008] PTL 1: Japanese Patent No. 5567227

PTL 2: Japanese Patent No. 5689543

PTL 3: Japanese Patent No. 6285043

PTL 4: Japanese Patent No. 6084711

SUMMARY OF INVENTION

TECHNICAL PROBLEM

[0009] An object of the present invention is to resolve the problem of particle generation in an Fe-Pt-BN-based sputtering target having a high relative density by an approach different from the inventions disclosed in PTL 1 to 4 above.

SOLUTION TO PROBLEM

[0010] The present inventors first confirmed that an Fe-Pt-BN-based sputtering target is difficult to attain a uniform fine microstructure due to the high melting point of BN whereas BN finely pulverized in such a uniform fine microstructure inhibits sintering of a composite material, resulting in poor sinterability. The present inventors then found possible to suppress particle generation by converting at least part of BN into boron oxide to lower the melting point and thus improving the sinterability of a BN-containing composite material, thereby completing the present invention.

[0011] According to the present invention, provided is an Fe-Pt-BN-based sputtering target containing nonmagnetic components of BN and boron oxide and having

a relative density of 88% or more,

an oxygen content of more than 4,000 wtppm and 10,000 wtppm or less, and

a percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) obtained by a procedure below of 1.0% or more.

(How to obtain a water-soluble boron concentration relative to a total boron

concentration)

The procedure includes:

(1) cutting out a 4 mm-square sample piece from the sputtering target and pulverizing the sample piece to prepare a pulverized product;

(2) classifying the pulverized product using sieves of 106 μm and 300 μm in opening size, immersing 0.50 g of a powder that has passed through the 300 μm sieve and remained on the 106 μm sieve in 100 mL of pure water at 25°C for 1 hour, and then filtering through a 5A filter paper specified in JIS P 3801;

(3) feeding the resulting filtrate into a 200 mL volumetric flask, filling up the flask, and obtaining a B concentration in the resulting solution through ICP analysis;

(4) obtaining a blank B concentration through ICP analysis of 200 mL of pure water alone;

(5) subtracting the blank B concentration from the B concentration in the solution and multiplying a liquid volume of 200 mL, thereby calculating the mass of dissolved water-soluble B;

(6) calculating a water-soluble B concentration per 1 g of the powder from the mass of water-soluble B in the powder mass of 0.50 g;

(7) weighing the powder on the 106 μm sieve in (2) above, subjecting to alkali fusion, and then calculating a total B concentration in 1 g of the powder through ICP analysis; and

(8) dividing the water-soluble B concentration in 1 g of the powder by the total B concentration in 1 g of the powder.

[0012] Moreover, according to the present invention, provided is an Fe-Pt-BN-based sputtering target containing nonmagnetic components of BN, boron oxide, and carbon (C) and having

a relative density of 88% or more,

an oxygen content of more than 4,000 wtppm and 10,000 wtppm or less, and

a percentage of a water-soluble boron concentration (wt%) relative to a total boron

concentration (wt%) obtained by the above-mentioned procedure of 1.0% or more.

[0013] It is preferable that O is present in a region where B exists on a cross-section of the Fe-Pt-BN-based sputtering target.

[0014] The Fe-Pt-BN-based sputtering target preferably contains 10 mol% or more and 55 mol% or less of Pt.

[0015] The Fe-Pt-BN-based sputtering target preferably contains 10 mol% or more and 55 mol% or less of BN.

[0016] The Fe-Pt-BN-based sputtering target preferably further contains 0 mol% or more and 20 mol% or less of C.

[0017] The Fe-Pt-BN-based sputtering target may further contain one or more elements selected from Ag, Au, Co, Cr, Cu, Ge, Ir, Ni, Pd, Rh, and Ru.

[0018] Further, according to the present invention, a production method for the Fe-Pt-BN-based sputtering target is provided. The production method of the present invention includes: preparing a raw material powder mixture through mixing of raw material powders in a stirred media mill at 100 rpm or more and 200 rpm or less for 2 hours or more and 6 hours or less in total in any embodiment of (A) to (E) below; and collecting, from the prepared raw material powder mixture, a powder that has passed through a sieve of 300 μm in opening size and sintering, where the method is characterized by oxidizing at least part of BN powder surface.

[0019] (A) an embodiment of first mixing BN powder alone in the stirred media mill in an atmosphere containing oxygen and/or moisture, then feeding the remaining raw material powders into the stirred media mill, subsequently replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(B) an embodiment of first pulverizing BN powder alone in the stirred media mill in an argon gas atmosphere and then bringing into contact with a gas containing oxygen and/or moisture, subsequently feeding the remaining raw material powders into the stirred media mill, then replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(C) an embodiment of mixing raw material powders in the stirred media mill in an argon gas atmosphere and then bringing the resulting mixed powder into contact with a gas containing oxygen and/or moisture

(D) an embodiment of mixing raw material powders in the stirred media mill in an argon gas atmosphere, suspending the mixing before a predetermined mixing time elapses to bring into contact with a gas containing oxygen and/or moisture, and then resuming the mixing

(E) an embodiment of mixing raw material powders in the stirred media mill in an atmosphere containing argon gas as well as oxygen and/or moisture

[0020] More specifically, when nonmagnetic components consist of BN and boron oxide, the above-mentioned raw material powder mixture is preferably prepared in any embodiment of (A1) to (E1) below.

(A1) an embodiment of first mixing BN powder alone in the stirred media mill in an atmosphere containing oxygen and/or moisture, then feeding Fe powder and Pt powder into the stirred media mill, subsequently replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(B1) an embodiment of first pulverizing BN powder alone in the stirred media mill in an argon gas atmosphere and then bringing into contact with a gas containing oxygen and/or moisture, subsequently feeding Fe powder and Pt powder into the stirred media mill, then replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(C1) an embodiment of mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere and then bringing the resulting mixed powder into contact with a gas containing oxygen and/or moisture

(D1) an embodiment of mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere, suspending the mixing before a predetermined mixing time elapses to bring into contact with a gas containing oxygen and/or moisture, and then resuming the mixing

(E1) an embodiment of mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an atmosphere containing argon gas as well as oxygen and/or moisture

[0021] Furthermore, when nonmagnetic components consist of BN, boron oxide, and carbon (C), the above-mentioned raw material powder mixture is preferably prepared in any embodiment of (A2) to (E2) below.

(A2) an embodiment of first mixing BN powder alone in the stirred media mill in an atmosphere containing oxygen and/or moisture, then feeding C powder, Fe powder, and Pt powder into the stirred media mill, subsequently replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(B2) an embodiment of first pulverizing BN powder alone in the stirred media mill in an argon gas atmosphere and then bringing into contact with a gas containing oxygen and/or moisture, subsequently feeding C powder, Fe powder, and Pt powder into the stirred media mill, then replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(C2) an embodiment of mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere and then bringing the resulting mixed powder into contact with a gas containing oxygen and/or moisture

(D2) an embodiment of mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere, suspending the mixing before a predetermined mixing time elapses to bring into contact with a gas containing oxygen and/or moisture, and then resuming the mixing

(E2) an embodiment of mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill in an atmosphere containing argon gas as well as oxygen and/or moisture

[0022] The sintering is preferably performed at a sintering temperature of 600°C or higher and 1200°C or lower and a sintering pressure of 30 MPa or more and 200 MPa or less.

ADVANTAGEOUS EFFECTS OF INVENTION

[0023] An Fe-Pt-BN-based sputtering target of the present invention has a relative density

of 88% or more and can reduce the number of particles to be generated during magnetron sputtering.

Further, since an Fe-Pt-BN-based sputtering target of the present invention can reduce the number of particles without adding SiO_2, AgCu alloy, AuCu alloy, or the like, the magnetic characteristics of a sputtered film is less affected.

BRIEF DESCRIPTION OF DRAWINGS

[0024] Fig. 1 is a schematic view of a microstructure in an Fe-Pt-BN-based sputtering target of the present invention.

Fig. 2 shows EPMA images for the Fe-Pt-BN-based sputtering target of Example 2.

Fig. 3 is a graph showing the relationship between the oxygen content and the percentage of a water-soluble boron concentration relative to a total boron concentration in the Examples and the Comparative Examples.

Fig. 4 is a graph showing the relationship between the number of particles and the percentage of a water-soluble boron concentration relative to a total boron concentration in the Examples and the Comparative Examples.

Fig. 5 is a graph showing the relationship between the number of particles and the oxygen content in the Examples and the Comparative Examples.

DESCRIPTION OF EMBODIMENTS

[0025] Hereinafter, the present invention will be described in detail with reference to the attached drawings. However, the present invention is by no means limited to these descriptions.

[0026] [First Embodiment]

An Fe-Pt-BN-based sputtering target of the first embodiment contains nonmagnetic components of BN and boron oxide and has a relative density of 88% or more, an oxygen content of more than 4,000 wtppm and 10,000 wtppm or less, and a percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) obtained by a procedure below of 1.0% or more.

(How to obtain a water-soluble boron concentration relative to a total boron concentration)

The procedure includes:

(1) cutting out a 4 mm-square sample piece from the sputtering target and pulverizing the sample piece to prepare a pulverized product;

(2) classifying the pulverized product using sieves of 106 μm and 300 μm in opening size, immersing 0.50 g of a powder that has passed through the 300 μm sieve and remained on the 106 μm sieve in 100 mL of pure water at 25°C for 1 hour, and then filtering through a 5A filter paper specified in JIS P 3801;

(3) feeding the resulting filtrate into a 200 mL volumetric flask, filling up the flask, and obtaining a B concentration in the resulting solution through ICP analysis;

(4) obtaining a blank B concentration through ICP analysis of 200 mL of pure water alone;

(5) subtracting the blank B concentration from the B concentration in the solution and multiplying a liquid volume of 200 mL, thereby calculating the mass of dissolved water-soluble B;

(6) calculating a water-soluble B concentration per 1 g of the powder from the mass of water-soluble B in the powder mass of 0.50 g;

(7) weighing the powder on the 106 μm sieve in (2) above, subjecting to alkali fusion, and then calculating a total B concentration in 1 g of the powder through ICP analysis; and

(8) dividing the water-soluble B concentration in 1 g of the powder by the total B concentration in 1 g of the powder.

[0027] An Fe-Pt-BN-based sputtering target of the present invention contains more than 4,000 wtppm and 10,000 wtppm or less and preferably more than 4,000 wtppm and 8,000 wtppm or less of oxygen and has the percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) of 1.0% or more, preferably 2.0% or more, and more preferably 2.0% or more and 10.0% or less. Since BN and B are insoluble in water, the detection of water-soluble boron in the percentage mentioned above constitutes the evidence that boron oxide, such as B_2O_3, is contained.

[0028] In other words, an Fe-Pt-BN-based sputtering target of the present invention contains boron oxide in an amount to satisfy the percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) of 1.0% or more, preferably 2.0% or more, and more preferably 2.0% or more and 10.0% or less.

[0029] When the oxygen content and the percentage of water-soluble boron relative to the total boron fall within the above-mentioned ranges, at least part of high-melting BN is replaced by low-melting boron oxide, thereby improving the sinterability of an Fe-Pt-BN-based composite material. As described in detail in the Examples and the Comparative Examples hereinafter, it is confirmed through EPMA analysis (analysis conditions will be described hereinafter) that O is present in a region where B exists on the cross-section of an Fe-Pt-BN-based sputtering target of the present invention. Since O is present in a region where B exists on a cross-section, the state in which at least part of BN is replaced by low-melting boron oxide is presumably the state in which boron oxide (B_2O_3) is formed on the surface of BN as illustrated in the schematic view of Fig. 1.

[0030] Further, an Fe-Pt-BN-based sputtering target of the present invention has a relative density of 88% or more and preferably a relative density of 90% or more. When the relative density is less than 88%, the practical use as a sputtering target is impossible.

[0031] An Fe-Pt-BN-based sputtering target of the present invention contains nonmagnetic components of BN and boron oxide. In the present invention, nonmagnetic components act as grain boundary materials that isolate metal components in a granular magnetic thin film formed by sputtering. An Fe-Pt-BN-based sputtering target of the present invention does not contain SiO_2 that causes particle generation.

[0032] An Fe-Pt-BN-based sputtering target of the present invention desirably contains 10 mol% or more and 55 mol% or less and preferably 15 mol% or more and 50 mol% or less of Pt. By setting the Pt content within these ranges, it is possible to satisfactorily maintain the magnetic characteristics of an Fe-Pt-based alloy after film deposition.

[0033] An Fe-Pt-BN-based sputtering target of the present invention desirably contains 10 mol% or more and 55 mol% or less, preferably 15 mol% or more and 50 mol% or less, and

more preferably 20 mol% or more and 45 mol% or less of BN. Within these ranges, BN acts as a grain boundary material in a granular magnetic thin film of a magnetic recording medium.

[0034] An Fe-Pt-BN-based sputtering target of the present invention may further contain one or more metal elements selected from Ag, Au, Co, Cr, Cu, Ge, Ir, Ni, Pd, Rh, and Ru. The total amount of these additional metal elements may be set to 0 mol% or more and 20 mol% or less and preferably 0 mol% or more and 15 mol% or less based on the entire Fe-Pt-BN-based sputtering target. Within these ranges, it is possible to satisfactorily maintain the magnetic characteristics of an Fe-Pt-based alloy.

[0035] An Fe-Pt-BN-based sputtering target of the present invention can be produced by a method including: preparing a raw material powder mixture by mixing BN powder, Fe powder, and Pt powder in a stirred media mill at 100 rpm or more and 200 rpm or less for 2 hours or more and 6 hours or less in total; and collecting, from the prepared raw material powder mixture, a powder that has passed through a sieve of 300 μm in opening size and sintering, where the method is characterized by bringing at least BN powder into contact with a gas containing oxygen and/or moisture during the preparation of a raw material powder mixture, thereby preparing a raw material powder mixture in which at least part of BN surface has been oxidized.

[0036] An excessively small number of rotations in the stirred media mill is not preferable since uniform dispersing of BN is impossible, whereas an excessively large number of rotations is also not preferable since fine particles are formed to lower the relative density, thereby making the practical use as a sputtering target impossible. Moreover, an excessively short mixing time results in insufficient formation of water-soluble boron, whereas an excessively long mixing time results in sufficient formation of water-soluble boron but a high oxygen content. Consequently, desirable magnetic characteristics may not be attained. Further, an excessively high oxygen content could allow the oxidation of Fe as a main component to proceed to increase iron oxide, cause cracking or chipping during processing of an Fe-Pt-BN-based sintered compact into a sputtering target, and result in poor

machinability of the sintered compact.

[0037] In the production method of the present invention, an embodiment of contact between BN surface and a gas containing oxygen and/or moisture during mixing of raw material powders may optionally be selected from (A1) to (E1) below.

(A1) an embodiment of first mixing BN powder alone in the stirred media mill in an atmosphere containing oxygen and/or moisture, then feeding Fe powder and Pt powder into the stirred media mill, subsequently replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(B1) an embodiment of first pulverizing BN powder alone in the stirred media mill in an argon gas atmosphere and then bringing into contact with a gas containing oxygen and/or moisture, subsequently feeding Fe powder and Pt powder into the stirred media mill, then replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(C1) an embodiment of mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere and then bringing the resulting mixed powder into contact with a gas containing oxygen and/or moisture

(D1) an embodiment of mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere, suspending the mixing before a predetermined mixing time elapses to bring into contact with a gas containing oxygen and/or moisture, and then resuming the mixing

(E1) an embodiment of mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an atmosphere containing argon gas as well as oxygen and/or moisture

[0038] In the cases of embodiments (A1) to (E1), the amounts of oxygen and/or moisture contained within the stirred media mill or the amounts of oxygen and/or moisture to be brought into contact with the mixed powder are adjusted such that the mixed powder after stirring/mixing has an oxygen content of more than 4,000 wtppm and 10,000 wtppm or less. The amounts of oxygen and/or moisture can be adjusted according to the mass of a mixed powder or the volume of a stirred media mill by the oxygen partial pressure of an oxygen-

containing atmosphere and/or the dew point of a moisture-containing atmosphere.

[0039] In the case of embodiment (A1), the conditions for stirring BN powder alone in the stirred media mill in an oxygen-containing atmosphere are set to, for example, 1 hour or more and 3 hours or less at 100 rpm or more and 200 rpm or less as well as 2 hours or more and 6 hours or less in total together with a stirring/mixing time after feeding Fe powder and Pt powder. The oxygen-containing atmosphere for stirring BN powder alone may preferably be an argon gas atmosphere containing 30 vol% of oxygen, for example.

[0040] In the case of embodiment (B1), the conditions for stirring BN powder alone in the stirred media mill in an argon gas atmosphere need to avoid the formation of excessively fine BN powder and are thus set to, for example, 1 hour or more and 3 hours or less at 100 rpm or more and 200 rpm or less as well as 2 hours or more and 6 hours or less in total together with a stirring/mixing time after feeding Fe powder and Pt powder. An embodiment of bringing pulverized BN powder into contact with a gas containing oxygen and/or moisture may preferably be an embodiment of taking out the powder from the stirred media mill and spreading in a vat in the atmosphere for a predetermined time. By bringing fine particles of BN powder into contact with the atmosphere, the surface area of BN that comes into contact with oxygen increases compared with embodiment (C1). Consequently, boron oxide is readily formed through oxidation of BN surface.

[0041] In the case of embodiment (C1), after mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere, the resulting mixed powder may be brought into contact with a gas containing oxygen and/or moisture for 30 minutes or more, for example. The mixing conditions in the stirred media mill are set to, for example, 2 hours or more and 6 hours or less in total at 100 rpm or more and 200 rpm or less. An embodiment of bringing the mixed powder into contact with a gas containing oxygen and/or moisture may preferably be an embodiment of spreading the mixed powder in a vat in the atmosphere for a predetermined time in a similar manner to embodiment (B1).

[0042] Embodiment (D1) may include, for example, mixing BN powder, Fe powder, and Pt powder in the stirred media mill filled with argon gas, suspending the stirring/mixing once or

more in the middle of the stirring/mixing time, taking out the resulting mixed powder from the stirred media mill, bringing into contact with a gas containing oxygen and/or moisture for 30 minutes or more, and then stirring/mixing again in the stirred media mill filled with argon gas. The number of times the mixed powder is brought into contact with a gas containing oxygen and/or moisture in the middle of the mixing may be set to once or more and five times or less. The mixing conditions in the stirred media mill are set to, for example, 2 hours or more and 6 hours or less in total at 100 rpm or more and 200 rpm or less. An embodiment of bringing the mixed powder into contact with a gas containing oxygen and/or moisture may preferably be an embodiment of spreading the mixed powder in a vat in the atmosphere for a predetermined time in a similar manner to embodiments (B1) and (C1).

[0043] In the case of embodiment (E1), the conditions for mixing BN powder, Fe powder, and Pt powder in the stirred media mill in an atmosphere containing argon gas as well as oxygen and/or moisture are set to, for example, 2 hours or more and 6 hours or less at 100 rpm or more and 200 rpm or less. The atmosphere containing argon gas as well as oxygen and/or moisture may preferably be an argon gas atmosphere containing 30 vol% or oxygen, for example.

[0044] In the cases of mixing in embodiments (A1) to (E1) above, the percentage of a water-soluble boron concentration relative to a total boron concentration in an Fe-Pt-BN-based sputtering target represents the percentage of boron oxide formed through decomposition of BN. The decomposition of BN, in other words, the formation of boron oxide proceeds, after BN is pulverized first, through oxidation of B in contact with oxygen or moisture. Since the relative density decreases when BN is excessively pulverized, mild conditions are required for mixing in the stirred media mill to avoid excessive formation of boron oxide.

[0045] In the case of containing one or more metal elements selected from Ag, Au, Co, Cr, Cu, Ge, Ir, Ni, Pd, Rh, and Ru as additional components, metal element powder or alloy powder (hereinafter, referred to as "metal powder") containing these additional elements may be added together with Fe powder and Pt powder in any of embodiments (A1) to (E1) above.

[0046] As Fe powder, a powder having an average particle size of 1 μm or more and 10 μm or less is preferably used. An excessively small average particle size is not preferable since the risk of ignition or the concentration of incidental impurities is likely to increase. Meanwhile, an excessively large average particle size is also not preferable since uniform dispersing of BN is impossible.

[0047] As Pt powder, a powder having an average particle size of 0.1 μm or more and 10 μm or less is preferably used. An excessively small average particle size is not preferable since the concentration of incidental impurities is likely to increase. Meanwhile, an excessively large average particle size is also not preferable since uniform dispersing of BN is impossible.

[0048] As BN powder, a powder having an average particle size of 2 μm or more and 10 μm or less is preferably used. An average particle size outside this range is not preferable since a desirable dispersion state cannot be achieved.

[0049] As metal powders used as other additional components, powders having an average particle size of 0.1 μm or more and 20 μm or less are preferably used. An excessively small average particle size is not preferable since the concentration of incidental impurities is likely to increase. Meanwhile, an excessively large average particle size is also not preferable since uniform dispersing is impossible.

[0050] The sintering is desirably performed at a sintering temperature of 600°C or higher and 1,200°C or lower and preferably 700°C or higher and 1,100°C or lower and a sintering pressure of 30 MPa or more and 200 MPa or less and preferably 50 MPa or more and 100 MPa or less. An excessively low sintering temperature is not preferable since the relative density decreases. Meanwhile, an excessively high sintering temperature is also not preferable since there is the risk of decomposing BN more than boron oxide (B_2O_3) formed.

[0051] [Second Embodiment]

An Fe-Pt-BN-based sputtering target of the second embodiment contains nonmagnetic components of C, BN, and boron oxide and has a relative density of 88% or more, an oxygen content of more than 4,000 wtppm and 10,000 wtppm or less, and a

percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) obtained by the procedure described for the first embodiment of 1.0% or more.

[0052] An Fe-Pt-BN-based sputtering target of the second embodiment contains C as a nonmagnetic component in addition to BN and boron oxide of the first embodiment. In the same manner as the first embodiment, an Fe-Pt-BN-based sputtering target of the second embodiment does not contain SiO_2 that causes particle generation.

[0053] An Fe-Pt-BN-based sputtering target of the present invention desirably contains 10 mol% or more and 55 mol% or less, preferably 15 mol% or more and 50 mol% or less, and more preferably 20 mol% or more and 45 mol% or less of BN.

[0054] When C is contained as a nonmagnetic component, the total of BN and C is desirably 10 mol% or more and 55 mol% or less, preferably 15 mol% or more and 50 mol% or less, and more preferably 20 mol% or more and 45 mol% or less. In this case, C is desirably more than 0 mol% and 20 mol% or less and preferably more than 0 mol% and 15 mol% or less. Within these ranges, C together with BN acts as a grain boundary material in a granular magnetic thin film of a magnetic recording medium.

[0055] An Fe-Pt-BN-based sputtering target of the second embodiment is the same as an Fe-Pt-BN-based sputtering target of the first embodiment except for further containing C as a nonmagnetic material.

[0056] An Fe-Pt-BN-based sputtering target of the present invention desirably contains 10 mol% or more and 55 mol% or less and preferably 15 mol% or more and 50 mol% or less of Pt. By setting the Pt content within these ranges, it is possible to satisfactorily maintain the magnetic characteristics of an Fe-Pt-based alloy after film deposition.

[0057] An Fe-Pt-BN-based sputtering target of the present invention may further contain one or more metal elements selected from Ag, Au, Co, Cr, Cu, Ge, Ir, Ni, Pd, Rh, and Ru. The total amount of these additional metal elements may be set to 0 mol% or more and 20 mol% or less and preferably 0 mol% or more and 15 mol% or less based on the entire Fe-Pt-BN-based sputtering target. Within these ranges, it is possible to satisfactorily maintain the

magnetic characteristics of an Fe-Pt-based alloy.

[0058] An Fe-Pt-BN-based sputtering target of the second embodiment can be produced by a method including: preparing a raw material powder mixture by mixing BN powder, C powder, Fe powder, and Pt powder in a stirred media mill at 100 rpm or more and 200 rpm or less for 2 hours or more and 6 hours or less in total; and collecting, from the prepared raw material powder mixture, a powder that has passed through a sieve of 300 μm in opening size and sintering, where the method is characterized by bringing at least BN surface into contact with a gas containing oxygen and/or moisture to oxidize at least partially during the preparation of a raw material powder mixture.

[0059] The above-mentioned raw material powder mixture may be prepared in any embodiment of (A2) to (E2) below.

(A2) an embodiment of first mixing BN powder alone in the stirred media mill in an atmosphere containing oxygen and/or moisture, then feeding C powder, Fe powder, and Pt powder into the stirred media mill, subsequently replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(B2) an embodiment of first pulverizing BN powder alone in the stirred media mill in an argon gas atmosphere and then bringing into contact with a gas containing oxygen and/or moisture, subsequently feeding C powder, Fe powder, and Pt powder into the stirred media mill, then replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, followed by mixing

(C2) an embodiment of mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere and then bringing the resulting mixed powder into contact with a gas containing oxygen and/or moisture

(D2) an embodiment of mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere, suspending the mixing before a predetermined mixing time elapses to bring into contact with a gas containing oxygen and/or moisture, and then resuming the mixing

(E2) an embodiment of mixing BN powder, C powder, Fe powder, and Pt powder in

the stirred media mill in an atmosphere containing argon gas as well as oxygen and/or moisture

[0060] In the cases of embodiments (A2) to (E2), the amounts of oxygen and/or moisture contained within the stirred media mill or the amounts of oxygen and/or moisture to be brought into contact with the mixed powder are adjusted such that the mixed powder after stirring/mixing has an oxygen content of more than 4,000 wtppm and 10,000 wtppm or less. The amounts of oxygen and/or moisture can be adjusted according to the mass of a mixed powder or the volume of a stirred media mill by the oxygen partial pressure of an oxygen-containing atmosphere and/or the dew point of a moisture-containing atmosphere.

[0061] In the case of embodiment (A2), the conditions for stirring BN powder alone in the stirred media mill in an oxygen-containing atmosphere are set to, for example, 1 hour or more and 3 hours or less at 100 rpm or more and 200 rpm or less as well as 2 hours or more and 6 hours or less in total together with a stirring/mixing time after feeding C powder, Fe powder, and Pt powder. The oxygen-containing atmosphere for stirring BN powder alone may preferably be an argon gas atmosphere containing 30 vol% of oxygen, for example.

[0062] In the case of embodiment (B2), the conditions for stirring BN powder alone in the stirred media mill in an argon gas atmosphere need to avoid the formation of excessively fine BN powder and are thus set to, for example, 1 hour or more and 3 hours or less at 100 rpm or more and 200 rpm or less as well as 2 hours or more and 6 hours or less in total together with a stirring/mixing time after feeding C powder, Fe powder, and Pt powder. An embodiment of bringing pulverized BN powder into contact with a gas containing oxygen and/or moisture may preferably be an embodiment of taking out the powder from the stirred media mill and spreading in a vat in the atmosphere for a predetermined time. By bringing fine particles of BN powder into contact with the atmosphere, the surface area of BN that comes into contact with oxygen increases compared with embodiment (C2). Consequently, boron oxide is readily formed through oxidation of BN surface.

[0063] In the case of embodiment (C2), after mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill in an argon gas atmosphere, the resulting mixed

powder is brought into contact with a gas containing oxygen and/or moisture for 30 minutes or more, for example. The mixing conditions in the stirred media mill are set to, for example, 2 hours or more and 6 hours or less in total at 100 rpm or more and 200 rpm or less. An embodiment of bringing the mixed powder into contact with a gas containing oxygen and/or moisture may preferably be an embodiment of spreading the mixed powder in a vat in the atmosphere for a predetermined time in a similar manner to embodiment (B2).

[0064] Embodiment (D2) may include, for example, mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill filled with argon gas, suspending the stirring/mixing once or more in the middle of the stirring/mixing time, taking out the resulting mixed powder from the stirred media mill, bringing into contact with a gas containing oxygen and/or moisture for 30 minutes or more, and then stirring/mixing again in the stirred media mill filled with argon gas. The number of times the mixed powder is brought into contact with a gas containing oxygen and/or moisture in the middle of the mixing may be set to once or more and five times or less. The mixing conditions in the stirred media mill are set to, for example, 2 hours or more and 6 hours or less in total at 100 rpm or more and 200 rpm or less. An embodiment of bringing the mixed powder into contact with a gas containing oxygen and/or moisture may preferably be an embodiment of spreading the mixed powder in a vat in the atmosphere for a predetermined time in a similar manner to embodiments (B2) and (C2).

[0065] In the case of embodiment (E2), the conditions for mixing BN powder, C powder, Fe powder, and Pt powder in the stirred media mill in an atmosphere containing argon gas as well as oxygen and/or moisture are set to, for example, 2 hours or more and 6 hours or less at 100 rpm or more and 200 rpm or less. The atmosphere containing argon gas as well as oxygen and/or moisture is preferably an argon gas atmosphere containing 30 vol% of oxygen and/or moisture.

[0066] In the cases of mixing in embodiments (A2) to (E2) above, the percentage of a water-soluble boron concentration relative to a total boron concentration in an Fe-Pt-BN-based sputtering target represents the percentage of boron oxide formed through

decomposition of BN. The decomposition of BN, in other words, the formation of boron oxide proceeds, after BN is pulverized first, through oxidation of B in contact with oxygen or moisture. Since the relative density decreases when BN is excessively pulverized, mild conditions are required for mixing in the stirred media mill to avoid excessive formation of boron oxide.

[0067] In the case of containing one or more metal elements selected from Ag, Au, Co, Cr, Cu, Ge, Ir, Ni, Pd, Rh, and Ru as additional components, metal element powder or alloy powder (hereinafter, referred to as "metal powder") containing these additional elements may be added together with Fe powder and Pt powder in any of embodiments (A2) to (E2) above.

[0068] As C powder, a powder having an average particle size of 2 μm or more and 10 μm or less is preferably used. An average particle size outside this range is not preferable since a desirable dispersion state cannot be achieved.

[0069] BN powder, Fe powder, Pt powder, other additional components, and sintering conditions are as described for the first embodiment, and hence, redundant explanation will be omitted.

EXAMPLES

[0070] Hereinafter, the present invention will be described specifically by means of Examples and Comparative Examples. For each sputtering target in the Examples and Comparative Examples below, the methods of measuring the relative density, the number of particles, the oxygen content, and the percentage of a water-soluble boron concentration relative to a total boron concentration are as follows.

[0071] [Relative Density]

The density is measured by the Archimedes method using pure water as a replacement liquid. An actual density (g/cm^3) is determined by measuring the mass of a sintered compact; measuring the buoyant force of the sintered compact (= volume of sintered compact) in the state of floating on the replacement liquid; and dividing the mass (g) of the sintered compact by the volume (cm^3) of the sintered compact. The percentage (actual density/theoretical density × 100) to a theoretical density that is calculated on the basis of the

composition of the sintered compact is a relative density.

[0072] [Number of Particles]

A sintered compact prepared under the mixing conditions and the sintering conditions shown in Table 2 is processed into a diameter of 153 mm and a thickness of 2 mm and bonded using indium to a Cu backing plate having a diameter of 161 mm and a thickness of 4 mm to yield a sputtering target. The sputtering target is fixed to a magnetron sputtering apparatus. After discharging in an Ar gas atmosphere at an output of 500 W and a gas pressure of 1 Pa for 4 hours, the number of particles adhered onto a substrate during sputtering for 40 seconds is determined by a particle counter.

[0073] [Oxygen Content]

(1) A 2 mm-square sample piece is cut out from a sputtering target using a bolt clipper, 0.1 g of an analysis sample is weighed out, and

(2) the oxygen content is measured with an oxygen/nitrogen analyzer under conditions of the highest temperature of 2,684°C.

[0074] [Percentage of Water-soluble Boron Concentration relative to Total Boron Concentration]

The procedure includes:

(1) cutting out a 4 mm-square sample piece from a sputtering target using a bolt clipper and pulverizing the sample piece to prepare a pulverized product;

(2) classifying the pulverized product using sieves of 106 μm and 300 μm in opening size, immersing 0.50 g of a powder that has passed through the 300 μm sieve and remained on the 106 μm sieve in 100 mL of pure water at 25°C for 1 hour, and then filtering through a 5A filter paper specified in JIS P 3801;

(3) feeding the resulting filtrate into a 200 mL volumetric flask, filling up the flask, and obtaining a B concentration in the resulting solution through ICP analysis;

(4) obtaining a blank B concentration through ICP analysis of 200 mL of pure water alone;

(5) subtracting the blank B concentration from the B concentration in the solution

and multiplying a liquid volume of 200 mL, thereby calculating the mass of dissolved water-soluble B;

(6) calculating a water-soluble B concentration per 1 g of the powder from the mass of water-soluble B in the powder mass of 0.50 g;

(7) placing 0.15 g of the powder that has remained on the 106 μm sieve in (2) above, 0.5 g of Na_2CO_3 powder (special grade sodium carbonate from Kanto Chemical Co., Inc.), and 2.0 g of Na_2O_2 powder (first grade cica-reagent sodium peroxide from Kanto Chemical Co., Inc.) in a zirconium crucible;

(8) subjecting to alkali fusion under conditions of heating at 700°C for 50 seconds, then heating at 900°C for 200 seconds, and further heating at 900°C for 200 seconds while rotating the crucible;

(9) immersing the crucible after alkali fusion in a mixed solution of 40 mL of pure water and 30 mL of hydrochloric acid (special grade hydrochloric acid from Kanto Chemical Co., Inc.) for 1 hour;

(10) feeding the resulting solution into a 100 mL volumetric flask, filling up the flask, diluting the solution 25 times, obtaining a B concentration in the resulting solution through ICP analysis, and calculating a total B concentration per 1 g of the powder; and

(11) dividing the water-soluble B concentration per 1 g of the powder by the total B concentration per 1 g of the powder.

[0075] [EPMA Analysis]

A 2 mm-square sample piece is cut out from a sputtering target, polished with a SiC abrasive paper of 1200 in particle size, and then buffed with a diamond spray of 1 μm. The resulting sample piece was analyzed with an EPMA apparatus (JXA-8500F) under the EPMA analysis settings shown in Table 1.

[0076] [Table 1]

Accelerating Voltage	15.0 kV		
Probe Current	1.5E-7 A		
Dwell Time	60.0 msec		
Number of Pixels	X: 128 Y: 128		
Pixel Size	X: 0.1564 μm Y: 0.1564 μm		
Element	B	N	O
X-ray Source	Kα	Kα	Kα
Analyzing Crystal	LDE2H	LDE1H	LDE1
Spect. Pos.	189.526	147.948	109.983
PHA Gain	32	64	64
High Volt (V)	1758	1696	1670
Base Level (V)	0.7	0.7	0.7
Window (V)	9.3	9.3	9.3
Diff/Int	Diff	Diff	Diff

[0077] [Example 1]

To have the composition of Fe-31.5Pt-30BN (in mol%, Fe and incidental impurities for the balance; the same applies to the following Examples and Comparative Examples), 190.28 g of Fe powder having an average particle size of 7 μm, 543.83 g of Pt powder having an average particle size of 1 μm, and 65.90 g of BN powder having an average particle size of 4 μm were weighed, fed into a stirred media mill (media: zirconia balls), and, after replacing the atmosphere inside the stirred media mill by argon gas, mixed at 150 rpm for 4 hours. The resulting mixed powder was classified with a sieve of 300 μm in opening size, and a powder that had passed through the sieve was spread in a vat to come into contact with the atmosphere for 30 minutes, thereby forming boron oxide (B_2O_3) in the vicinity of BN particles in the mixed powder. The resulting powder was sintered under conditions of a sintering pressure of 66 MPa, a sintering temperature of 900°C, and a holding time of 1 hour to yield a sintered compact.

[0078] After the relative density was measured, the sintered compact was processed into a sputtering target to measure the number of particles. Subsequently, a sample piece was cut out from the sputtering target to measure the oxygen content and the percentage of a water-soluble boron concentration relative to a total boron concentration. The relative density was 93.8%, the number of particles was 53, the percentage of a water-soluble boron concentration relative to a total boron concentration was 3.6%, and the oxygen content was 6,275 wtppm.

[0079] [Example 2]

To have the composition of Fe-30Pt-30BN-10C, 143.73 g of Fe powder having an average particle size of 7 μm, 502.08 g of Pt powder having an average particle size of 1 μm, 63.88 g of BN powder having an average particle size of 4 μm, and 10.30 g of C powder having an average particle size of 3 μm were weighed, fed into a stirred media mill (media: zirconia balls), and, after replacing the atmosphere inside the stirred media mill by argon gas, mixed at 150 rpm for 4 hours. The resulting mixed powder was classified with a sieve of 300 μm in opening size, and a powder that had passed through the sieve was spread in a vat to come into contact with the atmosphere for 30 minutes, thereby forming B_2O_3 in the vicinity of BN particles in the mixed powder. The resulting powder was sintered under conditions of a sintering pressure of 66 MPa, a sintering temperature of 900°C, and a holding time of 1 hour to yield a sintered compact.

[0080] After the relative density was measured, the sintered compact was processed into a sputtering target to measure the number of particles. Subsequently, a sample piece was cut out from the sputtering target to measure the oxygen content and the percentage of a water-soluble boron concentration relative to a total boron concentration. The relative density was 92.9%, the number of particles was 38, the percentage of a water-soluble boron concentration relative to a total boron concentration was 3.1%, and the oxygen content was 6,033 wtppm.

[0081] Further, a 2 mm-square sample piece was cut out from the sputtering target and subjected to EPMA analysis. The results are shown in Fig. 2. Fig .2 reveals that the regions where B exists are the same as the regions where N exists and that the regions where O is present overlap the regions where B and N exist but not the regions where Fe or Pt exists. Accordingly, it is presumed that O is present in regions where BN exists and forms boron oxide.

[0082] [Example 3]

A sintered compact was obtained and evaluated in the same manner as Example 2 except for changing the mixing conditions of raw material powders to 2 hours at 150 rpm. The relative density was 95.6%, the number of particles was 83, the percentage of a water-soluble boron concentration was 1.8%, and the oxygen content was 4,106 wtppm.

[0083] [Example 4]

First, 100.00 g of BN powder having an average particle size of 4 μm alone was fed into a stirred media mill (media: zirconia balls) and, after replacing the atmosphere inside the stirred media mill by a mixed gas atmosphere of 30 vol% of O_2 and Ar gas, mixed at 150 rpm for 2 hours.

[0084] Subsequently, to have the composition of Fe-30Pt-30BN-10C, 143.73 g of Fe powder having an average particle size of 7 μm, 502.08 g of Pt powder having an average particle size of 1 μm, 63.88 g of BN powder pulverized as above, and 10.30 g of C powder having an average particle size of 3 μm were weighed, fed into a stirred media mill (media: zirconia balls), and, after replacing the atmosphere inside the stirred media mill by argon gas, mixed at 150 rpm for 2 hours to prepare a raw material powder mixture.

The resulting raw material powder mixture was sintered under conditions of a sintering pressure of 66 MPa, a sintering temperature of 900°C, and a holding time of 1 hour to yield a sintered compact.

[0085] For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured in the same manner as Example 2. The relative density was 94.8%, the number of particles was 57, the percentage of a water-soluble boron concentration was 3.5%, and the oxygen content was 5,665 wtppm.

[0086] [Example 5]

First, 100.00 g of BN powder having an average particle size of 4 μm alone was fed into a stirred media mill (media: zirconia balls) and, after replacing the atmosphere inside the stirred media mill by an argon gas atmosphere, pulverized through mixing at 150 rpm for 2 hours. The resulting BN powder was spread in a vat to come into contact with the atmosphere for 30 minutes, thereby forming boron oxide (B_2O_3) in the vicinity of BN particles in the mixed powder.

[0087] Subsequently, to have the same composition as Example 4, 143.73 g of Fe powder having an average particle size of 7 μm, 502.08 g of Pt powder having an average particle

size of 1 μm, 63.88 g of BN powder pulverized as above, and 10.30 g of C powder having an average particle size of 3 μm were weighed, fed into a stirred media mill (media: zirconia balls), and, after replacing the atmosphere inside the stirred media mill by argon gas, mixed at 150 rpm for 2 hours to prepare a raw material powder mixture.

The resulting raw material powder mixture was sintered under conditions of a sintering pressure of 66 MPa, a sintering temperature of 900°C, and a holding time of 1 hour to yield a sintered compact.

[0088] For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured in the same manner as Example 2. The relative density was 94.0%, the number of particles was 48, the percentage of a water-soluble boron concentration was 5.4%, and the oxygen content was 7,706 wtppm.

[0089] [Example 6]

To have the same composition as Example 4, raw material powders of BN powder, C powder, Fe powder, and Pt powder were weighed, fed into a stirred media mill, and, after replacing the atmosphere inside the stirred media mill by an argon gas atmosphere containing 30 vol% of oxygen, mixed at 150 rpm for 2 hours to prepare a raw material powder mixture.

The resulting raw material powder mixture was sintered under conditions of a sintering pressure of 66 MPa, a sintering temperature of 900°C, and a holding time of 1 hour to yield a sintered compact.

[0090] For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured in the same manner as Example 2. The relative density was 92.9%, the number of particles was 78, the percentage of a water-soluble boron concentration was 2.9%, and the oxygen content was 4,638 wtppm.

[0091] [Example 7]

A sintered compact was obtained by preparing a raw material powder mixture in the same manner as Example 1 except for weighing, to have the composition of Fe-31.5Pt-7Ag-

30BN, 145.91 g of Fe powder having an average particle size of 7 μm, 509.70 g of Pt powder having an average particle size of 1 μm, 62.63 g of Ag powder having an average particle size of 10 μm, and 61.76 g of BN powder having an average particle size of 4 μm.

[0092] For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured in the same manner as Example 1. The relative density was 95.2%, the number of particles was 49, the percentage of a water-soluble boron concentration was 4.1%, and the oxygen content was 5,981 wtppm.

[0093] [Example 8]

A sintered compact was obtained by preparing a raw material powder mixture in the same manner as Example 1 except for weighing, to have the composition of Fe-31.5Pt-7Co-30BN, 151.43 g of Fe powder having an average particle size of 7 μm, 528.97 g of Pt powder having an average particle size of 1 μm, 35.51 g of Co powder having an average particle size of 3 μm, and 64.10 g of BN powder having an average particle size of 4 μm.

[0094] For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured in the same manner as Example 1. The relative density was 93.7%, the number of particles was 41, the percentage of a water-soluble boron concentration was 4.8%, and the oxygen content was 5,883 wtppm.

[0095] [Example 9]

A sintered compact was obtained by preparing a raw material powder mixture in the same manner as Example 1 except for weighing, to have the composition of Fe-31.5Pt-7Rh-30BN, 148.33 g of Fe powder having an average particle size of 7 μm, 518.15 g of Pt powder having an average particle size of 1 μm, 60.74 g of Rh powder having an average particle size of 10 μm, and 62.79 g of BN powder having an average particle size of 4 μm.

[0096] For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured in the same manner as Example 1. The relative density

was 92.5%, the number of particles was 43, the percentage of a water-soluble boron concentration was 3.8%, and the oxygen content was 6,121 wtppm.

[0097] [Example 10]

A sintered compact was obtained by preparing a raw material powder mixture in the same manner as Example 1 except for weighing, to have the composition of Fe-35Pt-30BN, 172.79 g of Fe powder having an average particle size of 7 μm, 603.60 g of Pt powder having an average particle size of 1 μm, and 65.83 g of BN powder having an average particle size of 4 μm, feeding into a stirred media mill, and mixing at 150 rpm for 3 hours. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 95.0%, the number of particles was 67, the percentage of a water-soluble boron concentration was 3.4%, and the oxygen content was 5,372 wtppm.

[0098] [Example 11]

A sintered compact was obtained by preparing a raw material powder mixture in the same manner as Example 10 except for weighing, to have the composition of Fe-32.5Pt-35BN, 157.91 g of Fe powder having an average particle size of 7 μm, 551.60 g of Pt powder having an average particle size of 1 μm, and 75.58 g of BN powder having an average particle size of 4 μm. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 94.1%, the number of particles was 77, the percentage of a water-soluble boron concentration was 3.3%, and the oxygen content was 4,833 wtppm.

[0099] [Example 12]

A sintered compact was obtained by preparing a raw material powder mixture in the same manner as Example 10 except for weighing, to have the composition of Fe-27.5Pt-45BN, 129.51 g of Fe powder having an average particle size of 7 μm, 452.40 g of Pt powder having an average particle size of 1 μm, and 94.19 g of BN powder having an average

particle size of 4 μm. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 91.4%, the number of particles was 94, the percentage of a water-soluble boron concentration was 2.8%, and the oxygen content was 5,296 wtppm.

[0100] [Example 13]

A sintered compact was obtained in the same manner as Example 2 except for feeding, to have the composition of Fe-35Pt-20BN-10C, 173.45 g of Fe powder having an average particle size of 7 μm, 605.89 g of Pt powder having an average particle size of 1 μm, 44.05 g of BN powder having an average particle size of 4 μm, and 10.66 g of C powder having an average particle size of 3 μm into a stirred media mill (media: zirconia balls) and mixing at 150 rpm for 3 hours. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 96.2%, the number of particles was 61, the percentage of a water-soluble boron concentration was 3.7%, and the oxygen content was 5,141 wtppm.

[0101] [Example 14]

A sintered compact was obtained in the same manner as Example 2 except for feeding, to have the composition of Fe-30Pt-30BN-10C, 143.73 g of Fe powder having an average particle size of 7 μm, 502.08 g of Pt powder having an average particle size of 1 μm, 63.88 g of BN powder having an average particle size of 4 μm, and 10.30 g of C powder having an average particle size of 3 μm into a stirred media mill (media: zirconia balls) as well as for changing the mixing conditions of raw material powders to 3 hours at 150 rpm. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 95.1%, the number of particles was 62, the percentage of a water-soluble boron concentration was 3.3%, and the oxygen content was 5,308 wtppm.

[0102] [Example 15]

A sintered compact was obtained in the same manner as Example 14 except for changing the sintering conditions of the raw material powder mixture to a sintering pressure of 66 MPa and a sintering temperature of 700°C. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 93.3%, the number of particles was 82, the percentage of a water-soluble boron concentration was 2.9%, and the oxygen content was 5,224 wtppm.

[0103] [Example 16]

A sintered compact was obtained in the same manner as Example 2 except for changing the mixing conditions of raw material powders to 6 hours at 150 rpm. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 90.7%, the number of particles was 33, the percentage of a water-soluble boron concentration was 7.3%, and the oxygen content was 7,688 wtppm.

[0104] [Example 17]

A sintered compact was obtained in the same manner as Example 2 except for feeding, to have the composition of Fe-25Pt-10Au-30BN-10C, 116.99 g of Fe powder having an average particle size of 7 μm, 408.33 g of Pt powder having an average particle size of 1 μm, 62.40 g of BN powder having an average particle size of 4 μm, 10.06 g of C powder having an average particle size of 3 μm, and 165.05 g of Au powder having an average particle size of 1 μm into a stirred media mill (media: zirconia balls) and mixing at 150 rpm for 3 hours. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 96.1%, the number of particles was 55, the percentage of a water-soluble boron concentration was 2.5%, and the oxygen content was 4,935 wtppm.

[0105] [Example 18]

A sintered compact was obtained in the same manner as Example 17 except for feeding, to have the composition of Fe-25Pt-10Ag-30BN-10C, 116.89 g of Fe powder having an average particle size of 7 μm, 408.33 g of Pt powder having an average particle size of 1 μm, 62.34 g of BN powder having an average particle size of 4 μm, 10.06 g of C powder having an average particle size of 3 μm, and 90.31 g of Ag powder having an average particle size of 10 μm into a stirred media mill (media: zirconia balls). For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 95.7%, the number of particles was 49, the percentage of a water-soluble boron concentration was 2.8%, and the oxygen content was 5,184 wtppm.

[0106] [Example 19]

A sintered compact was obtained in the same manner as Example 17 except for feeding, to have the composition of Fe-25Pt-10Cu-30BN-10C, 121.19 g of Fe powder having an average particle size of 7 μm, 423.33 g of Pt powder having an average particle size of 1 μm, 64.63 g of BN powder having an average particle size of 4 μm, 10.43 g of C powder having an average particle size of 3 μm, and 55.16 g of Cu powder having an average particle size of 3 μm into a stirred media mill (media: zirconia balls). For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 95.9%, the number of particles was 66, the percentage of a water-soluble boron concentration was 2.6%, and the oxygen content was 4,799 wtppm.

[0107] [Example 20]

A sintered compact was obtained in the same manner as Example 17 except for feeding, to have the composition of Fe-25Pt-10Rh-30BN-10C, 119.55 g of Fe powder having an average particle size of 7 μm, 417.61 g of Pt powder having an average particle size of 1 μm, 63.76 g of BN powder having an average particle size of 4 μm, 10.28 g of C powder having an average particle size of 3 μm, and 88.12 g of Rh powder having an average particle

size of 10 μm into a stirred media mill (media: zirconia balls). For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 94.0%, the number of particles was 88, the percentage of a water-soluble boron concentration was 2.8%, and the oxygen content was 5,041 wtppm.

[0108] [Example 21]

A sintered compact was obtained in the same manner as Example 17 except for feeding, to have the composition of Fe-25Pt-10Ge-30BN-10C, 112.65 g of Fe powder having an average particle size of 7 μm, 393.51 g of Pt powder having an average particle size of 1 μm, 60.08 g of BN powder having an average particle size of 4 μm, 9.69 g of C powder having an average particle size of 3 μm, and 58.61 g of Ge powder having an average particle size of 10 μm into a stirred media mill (media: zirconia balls) as well as for changing the sintering temperature to 700°C. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 97.0%, the number of particles was 60, the percentage of a water-soluble boron concentration was 2.3%, and the oxygen content was 5,222 wtppm.

[0109] [Comparative Example 1]

A sintered compact was obtained in the same manner as Example 2 except for changing the mixing conditions to 30 minutes at 150 rpm. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 95.4%, the number of particles was 563, the percentage of a water-soluble boron concentration was 0.2%, and the oxygen content was 1,318 wtppm.

[0110] [Comparative Example 2]

A sintered compact was obtained in the same manner as Example 2 except for changing the mixing conditions to 12 hours at 150 rpm. For the obtained sintered compact, the relative density, the percentage of a water-soluble boron concentration relative to a total

boron concentration, and the oxygen content were measured. The relative density was 87.4%, the percentage of a water-soluble boron concentration was 13.8%, and the oxygen content was 12,066 wtppm. The number of particles was not measured since the sintered compact is of little practical utility as a sputtering target due to the low relative density of 87.4%.

[0111] [Comparative Example 3]

A sintered compact was obtained in the same manner as Example 2 except for changing the mixing conditions to 5 minutes at 300 rpm. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 96.3%, the number of particles was 1,376, the percentage of a water-soluble boron concentration was 0.1%, and the oxygen content was 483 wtppm.

[0112] [Comparative Example 4]

A sintered compact was obtained in the same manner as Example 2 except for changing the mixing conditions to 30 minutes at 300 rpm. For the obtained sintered compact, the relative density, the number of particles, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 91.4%, the number of particles was 713, the percentage of a water-soluble boron concentration was 0.6%, and the oxygen content was 2,046 wtppm.

[0113] [Comparative Example 5]

A sintered compact was obtained in the same manner as Example 2 except for changing the mixing conditions to 2 hours at 300 rpm. For the obtained sintered compact, the relative density, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 86.5%, the percentage of a water-soluble boron concentration was 2.2%, and the oxygen content was 4,467 wtppm. The number of particles was not measured since the sintered compact is of little practical utility as a sputtering target due to the low relative density of 86.5%.

[0114] [Comparative Example 6]

A sintered compact was obtained in the same manner as Example 2 except for changing the mixing conditions to 6 hours at 460 rpm. For the obtained sintered compact, the relative density, the percentage of a water-soluble boron concentration relative to a total boron concentration, and the oxygen content were measured. The relative density was 79.2%, the percentage of a water-soluble boron concentration was 10.2%, and the oxygen content was 8,782 wtppm. The number of particles was not measured since the sintered compact is of little practical utility as a sputtering target due to the low relative density of 79.2%.

[0115] Fig. 3 shows the relationship between the oxygen content and the percentage of a water-soluble boron concentration relative to a total boron concentration in the Examples and the Comparative Examples. Fig. 4 shows the relationship between the number of particles generated and the percentage of a water-soluble boron concentration relative to a total boron concentration. Fig. 5 shows the relationship between the number of particles generated and the oxygen content.

[0116] Fig. 4 reveals that the number of particles exceeds 500 when the percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) falls within the range of less than 1.0% whereas the number of particles is extremely small as 100 or less when the percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) falls within the range of 1.0% or more.

[0117] Fig. 5 reveals that the number of particles exceeds 500 when the oxygen concentration falls within the range of less than 4,000 wtppm whereas the number of particles is extremely small as 100 or less when the oxygen concentration falls within the range of 4,000 wtppm or more and 10,000 wtppm or less.

[0118] From these results, it is found that Examples 1 to 21, in which the percentage of a water-soluble boron concentration (wt%) relative to a total boron concentration (wt%) is 1.0% or more and the oxygen content is more than 4,000 wtppm and 10,000 wtppm or less, have a relative density of 88% or more and the number of particles of less than 100 and thus

satisfy both the conditions of a high relative density and a small number of particles, whereas Comparative Examples 1 to 6, in which the above-mentioned requirements are not satisfied, have a relative density of less than 88% and thus cannot be put into practical use as a sputtering target (Comparative Examples 2, 5, and 6) or have a relative density of 88% or more but a considerably large number of particles as many as more than 500 (Comparative Examples 1, 3, and 4).

[0119] [Table 2]

Table 2 Raw Material Powder Mixing Conditions, Sintering Conditions, and Measured Results for Sintered Compacts (1/2)

	Composition	Mixing conditions									
	Composition	Raw material powder						BN powder mixing conditions		Raw material powder mixing conditions	
		Fe	Pt	BN	C	Others		Atmosphere	Mixing conditions	Atmosphere	Mixing conditions
	mol%	g	g	g	g		g				
Ex. 1	Fe-31.5Pt-30BN	190.28	543.83	65.90						Ar	150rpm4h
Ex. 2	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	150rpm4h
Ex. 3	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	150rpm2h
Ex. 4	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30			Ar-30%O_2	150rpm2h	Ar	150rpm2h
Ex. 5	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30			Ar	150rpm2h	Ar	150rpm2h
Ex. 6	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar-30%O_2	150rpm2h
Ex. 7	Fe-31.5Pt-7Ag-30BN	145.91	509.70	61.76		Ag: 10 μm	62.63			Ar	150rpm4h
Ex. 8	Fe-31.5Pt-7Co-30BN	151.43	528.97	64.10		Co: 3 μm	35.51			Ar	150rpm4h
Ex. 9	Fe-31.5Pt-7Rh-30BN	148.33	518.15	62.79		Rh: 10 μm	60.74			Ar	150rpm4h
Ex. 10	Fe-35Pt-30BN	172.79	603.60	65.83						Ar	150rpm3h
Ex. 11	Fe-32.5Pt-35BN	157.91	551.60	75.58						Ar	150rpm3h
Ex. 12	Fe-27.5Pt-45BN	129.51	452.40	94.19						Ar	150rpm3h
Ex. 13	Fe-35Pt-20BN-10C	173.45	605.89	44.05	10.66					Ar	150rpm3h
Ex. 14	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	150rpm3h
Ex. 15	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	150rpm3h
Ex. 16	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	150rpm6h
Ex. 17	Fe-25Pt-10Au-30BN-10C	116.99	408.33	62.40	10.06	Au: 1 μm	165.05			Ar	150rpm3h
Ex. 18	Fe-25Pt-10Ag-30BN-10C	116.89	408.33	62.34	10.06	Ag: 10 μm	90.31			Ar	150rpm3h
Ex. 19	Fe-25Pt-10Cu-30BN-10C	121.19	423.33	64.63	10.43	Cu: 3μm	55.16			Ar	150rpm3h
Ex. 20	Fe-25Pt-10Rh-30BN-10C	119.55	417.61	63.76	10.28	Rh: 10 μm	88.12			Ar	150rpm3h
Ex. 21	Fe-25Pt-10Ge-30BN-10C	112.65	393.51	60.08	9.69	Ge: 10 μm	58.61			Ar	150rpm3h
Comp. Ex. 1	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	150rpm30min
Comp. Ex. 2	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	150rpm12h
Comp. Ex. 3	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	300rpm5min
Comp. Ex. 4	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	300rpm30min
Comp. Ex. 5	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	300rpm2h
Comp. Ex. 6	Fe-30Pt-30BN-10C	143.73	502.08	63.88	10.30					Ar	460rpm6h

Table 2 Raw Material Powder Mixing Conditions, Sintering Conditions, and Measured Results for Sintered Compacts (2/2)

	Sintering conditions		Relative density	Number of particles	Water-soluble B concentration/total B concentration × 100	Oxygen concentration
	Pressure	Temperature				
	(MPa)	(°C)	(%)	(-)	(%)	(ppm)
Ex. 1	66	900	93.8	53	3.6	6275
Ex. 2	66	900	92.9	38	3.1	6033
Ex. 3	66	900	95.6	83	1.8	4016
Ex. 4	66	900	94.8	57	3.5	5665
Ex. 5	66	900	94.0	48	5.4	7706
Ex. 6	66	900	92.9	78	2.9	4638
Ex. 7	66	900	95.2	49	4.1	5981
Ex. 8	66	900	93.7	41	4.8	5883
Ex. 9	66	900	92.5	43	3.8	6121
Ex. 10	66	900	95.0	67	3.4	5372
Ex. 11	66	900	94.1	77	3.3	4833
Ex. 12	66	900	91.4	94	2.8	5296
Ex. 13	66	900	96.2	61	3.7	5141
Ex. 14	66	900	95.1	62	3.3	5308
Ex. 15	66	700	93.3	82	2.9	5224
Ex. 16	66	900	90.7	33	7.3	7688
Ex. 17	66	900	96.1	55	2.5	4935
Ex. 18	66	900	95.7	49	2.8	5184
Ex. 19	66	900	95.9	66	2.6	4799
Ex. 20	66	900	94.0	88	2.8	5041
Ex. 21	66	700	97.0	60	2.3	5222
Comp. Ex. 1	66	900	95.4	563	0.2	1318
Comp. Ex. 2	66	900	87.4	-	13.8	12066
Comp. Ex. 3	66	900	96.3	1376	0.1	483
Comp. Ex. 4	66	900	91.4	713	0.6	2046
Comp. Ex. 5	66	900	86.5	-	2.2	4467
Comp. Ex. 6	66	900	79.2	-	10.2	8782