

SPUTTERING TARGET MATERIAL, PRODUCTION METHOD
THEREFOR, AND THIN FILM PRODUCED THEREWITH

[Cross-Reference to Related Application]

5 [0001]

This application claims priority to Japanese Patent Application No. 2009-41817 filed on February 25, 2009, the entire content of which is incorporated herein by reference.

10 [Technical Field]

[0002]

The present invention relates to a sputtering target material for production of a Ni-W-Cr alloy intermediate layer film of a perpendicular magnetic recording medium, a method therefor,
15 and a thin film produced therewith.

[Background Art]

[0003]

In recent years, there have been remarkable progresses in
20 magnetic recording technology, and heightening of recording densities in magnetic recording media has been proceeding due to increasing drive capacities. However, in the magnetic recording media for longitudinal magnetic recording system conventionally used in the world, an attempt to realize a high
25 recording density results in refined recording bits, which require a high coercive force to such an extent that recording cannot be conducted with the recording bits. In view of this, perpendicular magnetic recording system has been investigated as a means for solving these problems and improving the
30 recording density.

[0004]

Perpendicular magnetic recording system is a system in which a magnetization-easy axis is oriented in the direction vertical to the medium surface of a magnetic film of a perpendicular
35 magnetic recording medium and is suitable for a high recording density. In addition, for perpendicular magnetic recording

system, a multi-layer recording medium having a magnetic recording film layer with an increased recording sensitivity, a soft magnetic film layer, and an intermediate layer has been developed. A CoCrPt-SiO₂-based alloy or the like is commonly
5 used for this magnetic recording film layer, while a Co-Zr-Nb-based alloy or the like is used for the soft magnetic film layer. The soft magnetic film layer plays a role in circulating a recording magnetic field from a magnetic head, having a role to improve read/write efficiency. Meanwhile, the
10 intermediate layer described herein generally refers to a layer which is provided for the purpose of refining the crystal grains in the magnetic recording film layer and imparting anisotropy to the crystal orientation.

[0005]

15 While various Ni-based alloys, Ta-based alloys, Pd-based alloys, Ru-based alloys and the like have been proposed for the intermediate layer, Ni-W-based alloys have been widely used in recent years. One of the roles of the intermediate layer is to control the structure of the magnetic recording film layer. For
20 this purpose, it is believed to be important to have crystallinity and refine the crystal grains. For instance, an example of a Ru intermediate layer has been proposed as disclosed in Japanese Patent Laid-Open Publication No. 2007-179598 (Patent Literature 1).

25 [0006]

In addition, as far as Ni-W-based alloy is concerned, the lattice constant of a thin film is thought to be satisfactory within the range of about 3.53 to 3.61 ($\times 10^{-10}$ m).

[Summary of Invention]

30 [0007]

However, although producing a perpendicular magnetic recording medium with a Ni-W-based thin film as an intermediate layer can provide good recording property, the recording bits need to be refined for achieving a higher
35 recording density, therefor necessitating refinement of the crystal grains in the Ni-W-based intermediate layer, which works

as an underlayer during formation of the magnetic recording film. Our analysis to date has revealed that addition of B or the like is effective for the refinement, which on the other hand involves disruption of the crystallinity to cause a problem in
5 maintaining the orientation of the magnetic recording film.

[0008]

The inventors have now found that addition of Cr to a Ni-W-based alloy makes it possible to drastically refine the crystal grain in the intermediate layer while retaining the
10 crystallinity.

[0009]

It is therefore an object of the present invention to provide a sputtering target material for producing an intermediate layer film of a perpendicular magnetic recording medium, in which
15 addition of Cr to a Ni-W-based alloy makes it possible to drastically refine the crystal grain in the intermediate layer while retaining the crystallinity, a method for producing the target material, and a thin film produced with the target material or by the production method.

20 [0010]

According to the present invention, there is provided a sputtering target material for use in production of an intermediate layer film of a perpendicular magnetic recording medium, wherein the sputtering target material is made of a
25 Ni-W-Cr alloy comprising in at%:

1 to 20% of W;

1 to 20% of Cr; and

balance Ni.

[0011]

30 According to the present invention, there is also provided a method for producing a sputtering target material for use in an intermediate layer film of a perpendicular magnetic recording medium, wherein the method comprises the steps of:

providing a raw material powder which provides an alloy
35 composition of a Ni-W-Cr alloy comprising, in at%, 1 to 20% of W; 1 to 20% of Cr; and balance Ni; and

consolidating the raw material powder.

[0012]

According to the present invention, there is further provided a Ni-W-Cr alloy thin film, which is produced by using the above
5 sputtering target material or the above method.

[Description of Embodiments]

[0013]

The present invention is described in detail below.

10 [0014]

The sputtering target material according to the present invention is to be used in production of the intermediate layer film of the perpendicular magnetic recording medium. The sputtering target material is made of a Ni-W-Cr alloy comprising,
15 in at%, 1 to 20% of W; 1 to 20% of Cr; and balance Ni.

[0015]

The sputtering target material of the present invention contains W in an amount of 1 to 20 at%, preferably 3 to 10 at%. W content of less than 1% results in an undesirable lattice
20 constant of the sputtered thin film being less than $3.53 (\times 10^{-10} \text{ m})$, while W content of more than 20% results in an undesirable lattice constant exceeding $3.61 (\times 10^{-10} \text{ m})$.

[0016]

The sputtering target material of the present invention contains
25 Cr in an amount of 1 to 20 at%, preferably 3 to 10 at%. Total Cr content of less than 1% fails to contribute to the crystal grain refinement in the sputtered thin film, while total Cr content of more than 20% makes the grain refinement effect level off to lead to a reduced effect of controlling orientation.

30 [0017]

The method for producing the sputtering target material according to the present invention comprises the steps of providing a raw material powder which provides an alloy composition of a Ni-W-Cr alloy comprising, in at%, 1 to 20% of
35 W; 1 to 20% of Cr; and balance Ni; and consolidating the raw material powder.

[0018]

It is preferable to use an alloy powder as the raw material powder for the following reasons. While Ni, Cr and W tend to form an alloy having a homogeneous composition of these three elements, allowing its crystal grains to enlarge in a melting process which is conducted at a low cooling rate. This causes an abnormal electrical discharge during sputtering to give rise to problems such as generation of many particles. In contrast, when a raw material powder is prepared by a gas atomization method, fine crystal grains are obtained since they have undergone rapid solidification. A sputtering target material consolidated with this raw material powder leads to a preferred reduction in particle generation.

[0019]

The consolidation temperature is preferred to be 800 to 1250°C. Consolidation at 800°C or higher makes the sintering sufficient to increase the relative density of the sputtering target material. In addition, consolidation at 1250°C or lower effectively prevents expansion of the billet during heating to enable more stable production.

[Examples]

[0020]

The present invention is explained in detail below with reference to examples.

[0021]

Ni-W-Cr alloy powders shown in Table 1 were prepared by gas atomization. The alloy powders were optionally mixed with one or more of pure metal powder of Ni, W and Cr to have the predetermined compositions, so as to be used as raw material powders. Powder-filled billets sealed in SC cans under evacuation were prepared by consolidating the raw material powders through HIP method or upset method at 750 to 1350°C, and were subjected to machining to prepare sputtering target materials of Ni-W-Cr alloys. Alternatively, Ni-W-Cr alloy sputtering target materials were also prepared by casting method. Each of these steps is explained in detail below.

[0022]

At the outset, 25 kg of molten raw material was subjected to induction melting in Ar in an alumina crucible and tapped at 1700°C through a tapping nozzle with a diameter of 5 mm positioned on the bottom part of the crucible, followed by Ar gas atomization at an atomizing pressure of 0.7 MPa to produce a powder. Depending on the necessity of adjusting the composition, the powder was mixed with one or more of pure metal powders of Ni, W and Cr that are prepared by similar gas atomization methods or are commercially available. The Ni-W-Cr alloy powders thus produced and mixed were each sealed in an SC can with an outer diameter of 205 mm, an inner diameter of 190 mm and a length of 300 mm under evacuation. The ultimate vacuum pressure during the evacuation was set at about 1.3×10^{-2} Pa.

[0023]

The powder-filled billet was consolidated by the HIP method at 900 to 1350°C and 147 MPa. Alternatively, the powder-filled billet was heated to 750 to 1200°C, and was then charged into a constraint-type container with a diameter of 215 mm, followed by consolidation at a pressure of 500 MPa. The consolidated materials produced by the above methods were processed to a disc with a diameter of 76.2 mm and a thickness of 3 mm by wire-cutting, lathe machining and flat surface polishing, and were attached to a copper backing plate by brazing to provide sputtering target materials.

[0024]

On the other hand, in the casting method, 100 kg of the molten raw material was melted under vacuum and casted into a mold with a diameter of 210 mm, which was then machined by a lathe to have a diameter of 200 mm and a length of 100 mm, followed by hot forging at 850°C to a height of 50 mm. The subsequent production method of the sputtering target material was conducted in the same manner as the above HIP and upset materials.

[0025]

The sputtering target materials thus produced were evaluated in accordance with the following evaluation items and methods. Billet expansion during consolidation was evaluated on the HIP material in terms of the appearance of the billet after the HIP.

- 5 The upset material was evaluated in terms of the appearance of the billet during its heating. The results were shown by denoting absence and presence of expansion as "good" and "poor," respectively.

[0026]

- 10 The relative density of the sputtering target material was determined by measuring the density based on the size and the weight of the disc with a diameter of 76.2 mm and a thickness of 3 mm, which was prepared by the above method, and calculating the ratio of the measured density to the theoretical
15 density calculated from the composition.

[0027]

- The number of particles in the sputtered film was evaluated by sputtering the sputtering target material toward a Si substrate with a diameter of 63.5 mm to form a sputtered film for
20 evaluation. The sputtering was conducted under conditions of an Ar pressure of 0.5 Pa and a DC power of 500 W. The thickness of the sputtered film was 500 nm. The number of the particles that generated at this time was measured. The numbers of particles in Table 1 are indicated as relative values
25 with regard to the number of particles of No. 1 being taken as 100.

[0028]

- The lattice constant of the sputtered film was determined by performing X-ray diffraction on the sputtered film and
30 calculating the lattice constant from the diffraction peak. By this X-ray diffraction, the width of the angles at which the intensity is half the peak intensity of a (111) plane was measured for evaluating crystallinity. The crystallinity constants in Table 1 are indicated as relative values with regard
35 to the crystallinity constant of No. 1 being taken as 100, meaning that smaller values indicate more crystallinity.

[0029]

The size of the crystal grain of the sputtered film was also confirmed. The crystal grain size of the sputtered film was measured by observing the cross-section of the sputtered film
5 by TEM and analyzing the TEM image to determine the diameter of a circle having an area corresponding to the area of the crystal grain, which was referred to as the crystal grain size. The sizes of the crystal grain in Table 1 are indicated as relative values with regard to the crystal grain size of No. 1 being taken
10 as 100, meaning that smaller values indicate smaller crystal grain sizes.

[0030][Table1]

Table 1

No	Composition (at%)			Consolidation Method	Consolidation Temperature (°C)	Expansion of Billet	Relative Density (%)	Number of Particles (Relative Value)	Lattice Constant ($\times 10^{-10}\text{m}$)	Crystallinity Constant (Relative Value)	Crystal Grain Size of Sputtered Film (Relative Value)
	W	Cr	Ni								
1	1	5	Bal.	Upset	1000	Good	99.8	100	3.53	100	100
2	8	5	Bal.	Upset	900	Good	99.4	120	3.56	120	110
3	20	5	Bal.	Upset	900	Good	99.2	180	3.61	140	140
4	2	5	Bal.	Upset	1100	Good	100	100	3.53	100	80
5	3	5	Bal.	Casting Method			99.6	150	3.54	110	90
6	10	5	Bal.	HIP	1050	Good	99.8	150	3.57	120	120
7	12	5	Bal.	HIP	1100	Good	100	160	3.58	120	130
8	5	1	Bal.	HIP	1000	Good	99.8	90	3.54	110	110
9	5	8	Bal.	Upset	900	Good	99.2	110	3.54	110	120
10	5	20	Bal.	HIP	900	Good	99.4	160	3.54	160	190
11	5	2	Bal.	Upset	1100	Good	100	100	3.54	110	110
12	5	3	Bal.	Upset	1000	Good	99.8	110	3.54	110	140
13	5	10	Bal.	HIP	1050	Good	100	140	3.54	120	140
14	5	12	Bal.	Upset	1100	Good	99.8	150	3.54	150	180
15	10	6	Bal.	HIP	800	Good	99.0	140	3.57	150	80
16	8	4	Bal.	HIP	1250	Good	100	170	3.56	140	120
17	12	5	Bal.	Casting Method			100	750	3.58	120	130
18	0	6	Bal.	HIP	800	Good	99.3	100	3.51	100	140
19	25	6	Bal.	HIP	1050	Good	99.7	300	3.62	180	150
20	8	0	Bal.	HIP	1000	Good	100	180	3.59	130	200
21	8	25	Bal.	HIP	1000	Good	100	210	3.59	240	180
22	1	5	Bal.	Upset	750	Good	97.1	150	3.53	100	90
23	1	5	Bal.	HIP	1350	Poor	Difficult to Investigate				

Nos. 1 to 17 shown in Table 1 are present invention examples, while Nos. 18 to 21 are comparative examples with Nos. 22 and 23 being reference examples.

[0031]

- 5 As shown in Table 1, comparative example No. 18 has a slightly low lattice constant due to the absence of W which is the mandatory component. Comparative example No. 19 has a slightly high lattice constant due to the high content of W which is the mandatory component. Comparative example No. 20 has
10 a large crystal grain size due to the absence of Cr which is the mandatory component. Comparison example No. 21 has a high relative value of particle number and a low crystallinity due to the high content of Cr which is the mandatory component.

[0032]

- 15 Reference example No. 22 has a low relative density due to the low consolidation temperature. Reference example No. 23 was difficult to investigate due to the high consolidation temperature which provided after HIP an expanded billet, which was difficult to be processed into a sputtering target material having an
20 density suitable for practical use. In contrast, present invention examples Nos. 1 to 17 all are found to satisfy the conditions of the present invention and thus are superior in each property.

[0033]

- 25 As described above, adding Cr to the conventional Ni-W binary components makes it possible to prepare a thin film with fine crystal grains, while retaining the crystallinity. Therefore, using the thin film as an intermediate layer to produce a perpendicular magnetic recording medium can result in a
30 satisfactory recording property.

CLAIMS

1. A sputtering target material for use in production of an intermediate layer film of a perpendicular magnetic recording medium, wherein the sputtering target material is made of a Ni-W-Cr alloy comprising in at%:
 - 1 to 20% of W;
 - 1 to 20% of Cr; and
 - balance Ni.
2. The sputtering target material according to claim 1, which is obtained by consolidating a powder having a composition of the alloy.
3. The sputtering target material according to claim 2, wherein the consolidation is conducted at a temperature from 800°C to 1250°C.
4. A Ni-W-Cr alloy thin film, which is produced by using the sputtering target material according to any one of claims 1 to 3.
5. A method for producing a sputtering target material for use in an intermediate layer film of a perpendicular magnetic recording medium, wherein the method comprises the steps of:
 - providing a raw material powder which provides an alloy composition of a Ni-W-Cr alloy comprising, in at%, 1 to 20% of W; 1 to 20% of Cr; and balance Ni; and
 - consolidating the raw material powder.
6. The method according to claim 5, wherein the consolidation is conducted at a temperature from 800°C to 1250°C.
7. A Ni-W-Cr alloy thin film, which is produced by using the method according to claim 5 or 6.