

CrTi-BASED ALLOY AND SPUTTERING TARGET MATERIAL,
PERPENDICULAR MAGNETIC RECORDING MEDIUM, AND
PRODUCTION METHODS THEREOF

5 [Cross-Reference to Related Application]

[0001]

This application claims priority to Japanese Patent Application No. 2010-182144 filed on August 17, 2010, the entire content of which is incorporated herein by reference.

10 [Technical Field]

[0002]

The present invention relates to a CrTi-based alloy and a sputtering target material with suppressed generation of compounds used for forming a thin film by sputtering and to
15 production methods thereof. The present invention also relates to a perpendicular magnetic recording medium produced using the CrTi-based alloy and the sputtering target material and to a production method thereof.

[Background Art]

20 [0003]

Generally, a CrTi-based target is used in a base film of a perpendicular magnetic recording medium and is obtained by hot-consolidating a pure Cr powder and a pure Ti powder. The CrTi-based target contains a high proportion of a brittle
25 compound phase, and the brittle compound phase generates particles during sputtering, which particles adhere to a sputtered film to decrease a product yield. Therefore, it is necessary to reduce compounds in a CrTi target.

[0004]

30 As a strategy for reducing compounds in such a CrTi target as described above, for example, a molten metal is quenched to reduce compounds, as disclosed in Japanese Patent Publication No. 64-2659 (Patent Literature 1). There is, however, a problem that a CrTi-based target is produced in a powder
35 metallurgy process and cannot be made into a molten metal.

[0005]

On the other hand, a powder sintered body is usually consolidated at a temperature of approximately 80% of its melting point. For example, a powder sintered body having a composition similar to a CrTi-based composition is consolidated at a temperature of 1200°C or more with hot press, as disclosed in Japanese Patent Laid-Open Publication No. 2003-226963 (Patent Literature 2). In addition, a powder sintered body is consolidated at a temperature of 1200°C by an upset method, as disclosed in Japanese Patent Laid-Open Publication No. 2002-212607 (Patent Literature 3). However, compounds tend to increase with a rise in the sintering temperature.

[0006]

Due to the high consolidating temperatures, Patent Literatures 2 and 3 both contain a problem that compound is abundant in a target, causing a generation of many particles during sputtering, thereby decreasing the product yield of a sputtered film.

[Citation List]

[0007]

[Patent Literature]

[PTL 1] Japanese Patent Publication No. 64-2659

[PTL 2] Japanese Patent Laid-Open Publication No. 2003-226963

[PTL 3] Japanese Patent Laid-Open Publication No. 2002-212607

[Summary of the Invention]

[0008]

The inventors have now found that a CrTi-based alloy and a sputtering target material capable of reducing particles to be generated in a sputtered film can be provided by reducing compounds in a sputtering target.

[0009]

It is therefore an object of the present invention to reduce compounds in a CrTi-based alloy and a sputtering target material, and thereby to suppress the generation of particles during sputtering to improve the product yield of a sputtered film.

[0010]

According to one embodiment of the present invention, there is

provided a CrTi-based alloy comprising 35-65 at% of Ti and the balance Cr and unavoidable impurities,

wherein the CrTi-based alloy has an intensity ratio $[I(\text{Cr}_2\text{Ti})/I(\text{Cr})]$ of an X-ray diffraction intensity $[I(\text{Cr}_2\text{Ti})]$ of Cr₂Ti(311) to an X-ray diffraction intensity $[I(\text{Cr})]$ of Cr(110), of 0.50 or less.

[0011]

According to another embodiment of the present invention, there is provided a CrTi-based sputtering target material comprising 35-65 at% of Ti and the balance Cr and unavoidable impurities,

wherein the sputtering target material has an intensity ratio $[I(\text{Cr}_2\text{Ti})/I(\text{Cr})]$ of an X-ray diffraction intensity $[I(\text{Cr}_2\text{Ti})]$ of Cr₂Ti(311) to an X-ray diffraction intensity $[I(\text{Cr})]$ of Cr(110), of 0.50 or less.

[0012]

According to another embodiment of the present invention, there is provided a perpendicular magnetic recording medium comprising a base film formed by sputtering using the CrTi-based sputtering target material.

[0013]

According to another embodiment of the present invention, there is provided a method for producing the CrTi-based alloy or the CrTi-based sputtering target material, the method comprising:

providing a raw material powder making up an overall composition of the alloy; and

hot-consolidating the raw material powder at 800 to 1100°C.

[0014]

According to another embodiment of the present invention, there is provided a method for producing the perpendicular magnetic recording medium, the method comprising:

providing a raw material powder making up an overall composition of the base film;

hot-consolidating the raw material powder at 800 to

1100°C to provide a CrTi-based sputtering target material; and forming the base film by sputtering using the CrTi-based sputtering target material.

[Description of Embodiments]

5 [0015]

The present invention is explained in detail below. The CrTi-based alloy, the CrTi-based sputtering target material, and the base film of the perpendicular magnetic recording medium, according to the present invention, are CrTi-based alloys comprising 35-65 at% of Ti and the balance Cr and unavoidable impurities, preferably consisting essentially of these components, and more preferably consisting of these components. The CrTi-based alloy and target material have an intensity ratio $[I(\text{Cr}_2\text{Ti})/I(\text{Cr})]$ of an X-ray diffraction intensity $[I(\text{Cr}_2\text{Ti})]$ of $\text{Cr}_2\text{Ti}(311)$ to an X-ray diffraction intensity $[I(\text{Cr})]$ of $\text{Cr}(110)$, of 0.50 or less.

[0016]

The CrTi-based alloy, the CrTi-based sputtering target material, and the base film of the perpendicular magnetic recording medium, according to the present invention, comprise 35 to 65 at%, preferably 40 to 60 at%, of Ti. When Ti is less than 35 at% or more than 65 at%, a sputtered film does not become amorphous in the case of using the alloy as a sputtering target material.

25 [0017]

The CrTi-based alloy, the CrTi-based sputtering target material, and the base film of the perpendicular magnetic recording medium, according to the present invention, have an intensity ratio $[I(\text{Cr}_2\text{Ti})/I(\text{Cr})]$ of an X-ray diffraction intensity $[I(\text{Cr}_2\text{Ti})]$ of $\text{Cr}_2\text{Ti}(311)$ to an X-ray diffraction intensity $[I(\text{Cr})]$ of $\text{Cr}(110)$, of 0.50 or less, preferably 0.07 or less, more preferably 0.03 or less. When the intensity ratio is higher than 0.50, many particles are generated.

[0018]

35 The CrTi-based alloy and the CrTi-based sputtering target material, according to the present invention, can be produced

by hot-consolidating a raw material powder at 800 to 1100°C, preferably at 800 to 1050°C. A hot-consolidating temperature of less than 800°C results in an insufficient density. On the other hand, a hot-consolidating temperature of more than
5 1100°C results in an increased X-ray diffraction intensity ratio value and the generation of many particles during sputtering, so that the particles adhere to a sputtered film to decrease a product yield. It is more preferable that the upper limit of a hot-consolidating temperature be 1000°C or less in an upset
10 method, or 900°C or less in an HIP method.

[0019]

According to a preferred embodiment of the present invention, an effect of suppressing the generation of compounds can be further enhanced by cooling an ingot from a hot consolidating
15 temperature at a cooling rate of 144 to 36000°C/hr after hot consolidating. That is, this is because, by the quenching at the above-described cooling rate, a CrTi solid solution in a high temperature phase is maintained to low temperature to suppress the transformation of the solid solution into a
20 compound. The preferred lower limit of the above-described cooling rate is 500°C/hr or more.

[0020]

The perpendicular magnetic recording medium according to the present invention can be produced through producing a
25 CrTi-based sputtering target material by hot-consolidating a raw material powder at 800 to 1100°C, preferably at 800 to 1050°C, in the same manner as described above and thereafter forming a base film by sputtering using the CrTi-based sputtering target material. The steps of producing the perpendicular magnetic
30 recording medium, other than the formation of the base film, are not particularly limited, and thus may appropriately adopt any conventional steps. According to a preferred embodiment of the present invention, due to the previously-described reason, it is preferable to, after the hot consolidating, cool the target
35 material from a consolidating temperature at a cooling rate of 144 to 36000°C/hr prior to the above-described sputtering, and,

in this case, the preferred lower limit of the above-described cooling rate is 500°C/hr or more.

[Examples]

[0021]

- 5 The present invention is specifically explained below with reference to Examples.

[0022]

- A pure Cr powder having a particle size of 250 μm or less was mixed with a pure Ti powder having a particle size of 150 μm or less in the formulation of Cr-Ti alloy composition shown in Table 1. The resultant mixed powder was charged into a sealed can made of steel material and was exhausted and vacuum-sealed at an ultimate vacuum of 10^{-1} Pa or more. Then, in the case of HIP (hot isostatic press), the powder was consolidated under conditions of a heating temperature of 800 to 1100°C, a consolidating pressure of 150 MPa, and a heating retention time of 1 hour, followed by producing ingots at a cooling rate controlled by air-cooling (Nos. 3, 4, 5, 7, 11, 12, 13, 15, 16, 19, 21, 22, 23, 25, 29, 30, 31, and 33) or water-cooling (Nos. 8, 9, 17, 26, 27, and 34) to 300°C under the conditions shown in Table 1. On the other hand, in the case of the upset method, the powder was consolidated under conditions of a heating temperature of 800 to 1100°C, a consolidating pressure of 500 MPa, and a heating retention time of 1 hour, followed by producing ingots at a cooling rate controlled by air-cooling or water-cooling to 300°C under the conditions shown in Table 1. The resultant ingots were then machined to produce targets.

[0023]

- The compound peak ratios $[I(\text{Cr}_2\text{Ti})/I(\text{Cr})]$ of the resultant targets were measured. The measurements were conducted by X-ray diffraction using Cu-K α ray as an X-ray source under a condition of a scan speed of 4°/min. For the evaluation of particles, films were formed on aluminum substrates having a diameter of 95 mm and a board thickness of 1.75 mm by DC magnetron sputtering at an Ar gas pressure of 0.9 Pa using the resultant targets to count the numbers of particles with an

optical surface analyzer. The results are shown in Table 1.

[0024]

[Table 1]

Table 1

No.	Consolidating Method	Composition (at%)	Consolidating Temperature (°C/hr)	Cooling Rate (°C/hr)	X-ray Diffraction Intensity [(Cr ₂ Ti)/I(Cr)]	Number of Particles	Evaluation	Present Invention Examples
1	Upset	Cr-35Ti	800	144	0.0232	10	Good	
2	Upset	Cr-45Ti	1000	144	0.0249	7	Good	
3	Upset	Cr-45Ti	1000	300	0.0186	5	Good	
4	Upset	Cr-45Ti	1000	500	0.0171	2	Good	
5	Upset	Cr-45Ti	1000	1500	0.0146	1	Good	
6	Upset	Cr-45Ti	1050	2500	0.149	15	Good	
7	Upset	Cr-35Ti	1050	144	0.138	14	Good	
8	Upset	Cr-35Ti	1050	36000	0.132	12	Good	
9	Upset	Cr-65Ti	800	36000	0.0250	8	Good	
10	Upset	Cr-50Ti	1000	144	0.0274	6	Good	
11	Upset	Cr-50Ti	1000	300	0.0262	4	Good	
12	Upset	Cr-50Ti	1000	500	0.0255	0	Good	
13	Upset	Cr-50Ti	1000	1500	0.0249	0	Good	
14	Upset	Cr-50Ti	1050	2500	0.365	14	Good	
15	Upset	Cr-65Ti	1050	144	0.413	11	Good	
16	Upset	Cr-50Ti	1100	300	0.450	15	Good	
17	Upset	Cr-65Ti	1050	36000	0.404	10	Good	
18	HIP	Cr-35Ti	800	144	0.0530	14	Good	
19	HIP	Cr-45Ti	900	500	0.0292	6	Good	
20	HIP	Cr-45Ti	1000	144	0.0636	14	Good	
21	HIP	Cr-45Ti	1000	500	0.0541	10	Good	
22	HIP	Cr-45Ti	1000	1500	0.0397	9	Good	
23	HIP	Cr-45Ti	1000	2500	0.0353	7	Good	
24	HIP	Cr-45Ti	1050	300	0.198	15	Good	
25	HIP	Cr-35Ti	1050	144	0.172	14	Good	
26	HIP	Cr-35Ti	1050	36000	0.132	13	Good	
27	HIP	Cr-65Ti	800	36000	0.0591	12	Good	
28	HIP	Cr-50Ti	1000	144	0.0689	11	Good	
29	HIP	Cr-50Ti	1000	500	0.0633	10	Good	
30	HIP	Cr-50Ti	1000	1500	0.0533	9	Good	
31	HIP	Cr-50Ti	1000	2500	0.0489	8	Good	
32	HIP	Cr-50Ti	1050	300	0.399	11	Good	
33	HIP	Cr-65Ti	1050	144	0.450	14	Good	
34	HIP	Cr-65Ti	1050	36000	0.438	13	Good	
35	HIP	Cr-50Ti	1100	500	0.500	14	Good	
36	Upset	Cr-35Ti	<u>700</u>	<u>80</u>	-	-	-	Comparative Examples
37	Upset	Cr-35Ti	<u>1200</u>	<u>80</u>	1.29	29	Poor	
38	Upset	Cr-65Ti	<u>700</u>	<u>80</u>	-	-	-	
39	Upset	Cr-65Ti	<u>1200</u>	<u>80</u>	1.48	24	Poor	
40	HIP	Cr-35Ti	<u>700</u>	<u>80</u>	-	-	-	
41	HIP	Cr-35Ti	<u>1200</u>	<u>80</u>	1.30	28	Poor	
42	HIP	Cr-65Ti	<u>700</u>	500	-	-	-	
43	HIP	Cr-65Ti	1000	<u>80</u>	1.58	26	Poor	
44	HIP	<u>Cr-30Ti</u>	1000	500	-	-	-	
45	HIP	<u>Cr-70Ti</u>	1050	600	-	-	-	

NOTE: The underlined figures fall outside the scope of the present invention.

As shown in Table 1, Nos. 1 to 35 are present invention examples, while Nos. 36 to 45 are comparative examples.

[0025]

Comparative Example Nos. 36, 38, and 40 shown in Table 1
5 were not evaluated, since the densities of the resultant powder ingots were low due to the low consolidating temperatures and the low cooling rates. All of Comparative Example Nos. 37, 39, and 41 resulted in high X-ray diffraction intensity ratio values and the large numbers of particles due to the high consolidating
10 temperatures and the low cooling rates. Comparative Example No. 42 was not evaluated since the density of the resultant powder ingot was low due to the low consolidating temperature. Comparative Example No. 43 results in a high X-ray diffraction intensity ratio value and the large number of particles due to
15 the low cooling rate after the consolidating.

[0026]

Comparative Example No. 44 was not evaluated since a film after sputtering did not become amorphous due to a low Ti content. Comparative Example No. 45 was not evaluated since
20 a film after sputtering did not become amorphous due to a high Ti content, similarly to Comparative Example No. 44. In contrast, it is found that, in Nos. 1 to 35, which are present invention examples, the X-ray diffraction intensities can be reduced to 0.5 or less and the numbers of particles are small
25 since all of them satisfy the conditions of the present invention.

[0027]

As described above, by hot-consolidating the raw material powder according to the present invention in the temperature range of 800 to 1100°C and optionally rapidly cooling it from
30 the consolidating temperature at a cooling rate of 144 to 36000°C/hr after the hot consolidating, a CrTi-based alloy and a CrTi-based target having a small amount of generated compounds were able to be produced to improve the product yield of a sputtered film.

35

CLAIMS

1. A CrTi-based alloy comprising 35-65 at% of Ti and the balance Cr and unavoidable impurities,
wherein the CrTi-based alloy has an intensity ratio $[I(\text{Cr}_2\text{Ti})/I(\text{Cr})]$ of an X-ray diffraction intensity $[I(\text{Cr}_2\text{Ti})]$ of $\text{Cr}_2\text{Ti}(311)$ to an X-ray diffraction intensity $[I(\text{Cr})]$ of $\text{Cr}(110)$, of 0.50 or less.
2. The CrTi-based alloy according to claim 1, consisting of 35-65 at% of Ti and the balance Cr and unavoidable impurities.
3. A CrTi-based sputtering target material comprising 35-65 at% of Ti and the balance Cr and unavoidable impurities,
wherein the sputtering target material has an intensity ratio $[I(\text{Cr}_2\text{Ti})/I(\text{Cr})]$ of an X-ray diffraction intensity $[I(\text{Cr}_2\text{Ti})]$ of $\text{Cr}_2\text{Ti}(311)$ to an X-ray diffraction intensity $[I(\text{Cr})]$ of $\text{Cr}(110)$, of 0.50 or less.
4. The CrTi-based sputtering target material according to claim 3, consisting of 35-65 at% of Ti and the balance Cr and unavoidable impurities.
5. A perpendicular magnetic recording medium comprising a base film formed by sputtering using the CrTi-based sputtering target material according to claim 3 or 4.
6. A method for producing the CrTi-based alloy or the CrTi-based sputtering target material according to any one of claims 1 to 4, the method comprising:
providing a raw material powder making up an overall composition of the alloy; and
hot-consolidating the raw material powder at 800 to 1100°C,
wherein the method further comprises cooling an ingot

from a consolidating temperature at a cooling rate of 144 to 36000°C/hr after the hot consolidating.

7. A method for producing the perpendicular magnetic recording medium according to claim 5, the method comprising:

- providing a raw material powder making up an overall composition of the base film;

- hot-consolidating the raw material powder at 800 to 1100°C to provide a CrTi-based sputtering target material; and

- forming the base film by sputtering using the CrTi-based sputtering target material,

wherein the method further comprises after the hot consolidating, cooling the target material from a consolidating temperature at a cooling rate of 144 to 36000°C/hr prior to the sputtering.