

## SPUTTERING TARGET MATERIAL

### BACKGROUND OF THE INVENTION

#### FIELD OF THE INVENTION

5 [0001]

The present invention relates to a sputtering target material used in a sputtering method for forming a metallic thin film.

#### 10 BACKGROUND ART

[0002]

Hard disks are used as external storages of computers, digital household appliances, and the like. Common hard disks adopt either in-plane recording or vertical magnetic recording  
15 format. In recent years, hard disks are required to have improved recording density. From this viewpoint, a vertical magnetic recording format is mainly used. Vertical magnetic recording format hard disks can have higher recording density and be more stably magnetically recorded at the same time.

20 [0003]

Japan Patent No. 4499044 discloses a magnetic recording medium including a laminate and a recording film formed on the laminate. In the laminate, formation is made on a substrate made of glass, aluminum alloy, or the like. The laminate  
25 comprises an adhesion layer, a soft magnetic under layer, a seed layer, and an intermediate layer. The adhesion layer is a film made of a Ni-Ta alloy. This film can be formed by a sputtering method.

[0004]

30 In a sputtering method, a target material is used. Sputtering target materials are manufactured by known methods, including casting methods such as vacuum melting and electron-beam melting, and sintering methods using mixed powders obtained by mixing multiple powders or using alloy  
35 powders. Japanese Patent Application Laid-Open (JP-A) No. 2000-206314 discloses a sintering method by hot isostatic

pressing.

## PRIOR ART DOCUMENTS

### PATENT DOCUMENTS

5 [0005]

Patent Document 1: Japan Patent No. 4499044

Patent Document 2: JP-A No. 2000-206314.

## SUMMARY OF THE INVENTION

### 10 PROBLEMS TO BE SOLVED BY THE INVENTION

[0006]

Conventionally used sputtering target materials have poor strength. Adhesion layers, which are relatively thick, need time to be sputtered. To shorten the time for formation of  
15 adhesion layers, a large amount of electric power may be used. Sputtering using a large amount of electric power results in a strain due to thermal stress in the sputtering target material. The strain can cause the sputtering target material to crack. Furthermore, a large amount of electric power can cause  
20 generation of particles in the sputtering target material.

[0007]

An object of the present invention is to provide a target material that is less likely to crack during sputtering.

### 25 SOLUTION TO THE PROBLEMS

[0008]

In one aspect of the present invention, provided are sputtering target materials as following [1] to [6]:

- [1] A sputtering target material consisting of an alloy,  
30 wherein the alloy consists of Ta and Cr with the balance being unavoidable impurities, and  
wherein the sputtering target material has a bending strength of 400 MPa or more as measured by a three-point bending test;  
35 [2] The sputtering target material according to [1], wherein the sputtering target material has a relative density of 97% or

more;

[3] The sputtering target material according to [1] or [2], wherein the sputtering target material has a Cr phase, a Ta phase, and a Cr<sub>2</sub>Ta phase;

5 [4] The sputtering target material according to [3], wherein the ratio (Px) of the intensity of Cr<sub>2</sub>Ta (220) to the intensity of Ta (110) in the X-ray diffraction (XRD) pattern is 10% or less;

[5] The sputtering target material according to [3] or [4], wherein the Cr<sub>2</sub>Ta phase is present on a surface of the Cr phase,  
10 and wherein the thickness (Tave) of said Cr<sub>2</sub>Ta phase is 30 μm or less; and

[6] The sputtering target material according to any one of [1] to [5], wherein the content of Ta is 20 at% or more and 90 at% or less, and wherein the content of Cr is 10 at% or more  
15 and 80 at% or less.

[0009]

In another aspect of the present invention, provided is a method of manufacturing the sputtering target material according to the present invention, including the following  
20 steps:

a step of mixing a powder made of Cr and a powder made of Ta to obtain a mixed powder; and

a step of applying a pressure to the mixed powder at a temperature of 1000°C or higher and 1350°C or lower.

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## EFFECT OF THE INVENTION

[0010]

The target material according to the present invention is less likely to crack during sputtering.

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## BRIEF DESCRIPTION OF THE DRAWING

[0011]

FIG. 1 is an enlarged image showing a part of the sputtering target material according to one embodiment of the  
35 present invention.

## DETAILED DESCRIPTION OF THE INVENTION

[0012]

Now, embodiments of the present invention will be described in detail by referring to the drawing as appropriate.

5 [0013]

The sputtering target material according to the present invention comprises or consists of the alloy according to the present invention. The alloy according to the present invention contains Ta and Cr. The balance of the alloy according to the present invention is unavoidable impurities. Examples of the unavoidable impurities include O, S, C, and N. In one embodiment, the alloy according to the present invention contains one or two or more unavoidable impurities. In a preferred embodiment, the content of O is 5,000 ppm or less, the content of S is 200 ppm or less, the content of C is 300 ppm or less, and the content of N is 300 ppm or less. The lower limit of the contents of O, S, C, and N are zero.

[0014]

The sputtering target material according to the present invention may be manufactured by so-called powder metallurgy. A method of manufacturing the sputtering target material includes mixing a powder made of Cr (i.e., an aggregate of Cr particles, each of which contains Cr with the balance being unavoidable impurities) and a powder made of Ta (i.e., an aggregate of Ta particles, each of which contains Ta with the balance being unavoidable impurities) to obtain a mixed powder. The mixed powder contains many particles. The mixed powder is pressurized at a high temperature. This pressurization allows the particles to be sintered with one another to provide a sputtering target material.

[0015]

Preferably, the sputtering target material according to the present invention has a Cr phase, a Ta phase, and a Cr<sub>2</sub>Ta phase. The Cr phase is derived from particles made of Cr. The Ta phase is derived from particles made of Ta. Cr<sub>2</sub>Ta is an intermetallic compound. This intermetallic compound is

produced by a reaction between Cr and Ta.

[0016]

The sputtering target material having a Cr phase, a Ta phase, and a Cr<sub>2</sub>Ta phase can provide a film having excellent properties. In the sputtering target material having a Cr phase, a Ta phase, and a Cr<sub>2</sub>Ta phase, the Ta phase can reduce a stress during sputtering. Thus, the content of Ta in the sputtering target material according to the present invention is preferably 20 at% or more and 90 at% or less, more preferably 30 at% or more and 80 at% or less. The content of Cr in the sputtering target material according to the present invention is preferably 10 at% or more and 80 at% or less, more preferably 20 at% or more and 70 at% or less.

[0017]

FIG. 1 is an enlarged image showing a part of the sputtering target material 2 according to one embodiment of the present invention. The image shown in FIG. 1 is obtained from an energy dispersive X-ray (EDX) analysis. EDX observes backscattered electron images. As shown in FIG. 1, the sputtering target material 2 has a Cr phase 4, a Ta phase 6, and a Cr<sub>2</sub>Ta phase 8.

[0018]

The Cr<sub>2</sub>Ta phase 8 exists at the interface between the Cr phase 4 and the Ta phase 6. The Cr<sub>2</sub>Ta phase 8 is present on the surface of the Cr phase 4 and is adhered to the surface of the Cr phase 4. The Cr<sub>2</sub>Ta phase 8 is brittle. The sputtering target material 2 is manufactured by sintering a mixed powder obtained by mixing a Cr powder (i.e., an aggregate of Cr particles, each of which contains Cr with the balance being unavoidable impurities) and a Ta powder (i.e., an aggregate of Ta particles, each of which contains Ta with the balance being unavoidable impurities). Therefore, Cr<sub>2</sub>Ta phase 8 production in the sputtering target material 2 can be reduced as compared to that in a sputtering target material obtained by a casting method. The sputtering target material 2 having less Cr<sub>2</sub>Ta phase 8 shows higher bending strength. From the viewpoint of

bending strength, the thickness ( $T_{ave}$ ) of the  $Cr_2Ta$  phase 8 is preferably 30  $\mu m$  or less, more preferably 25  $\mu m$  or less, still more preferably 20  $\mu m$  or less. The ideal thickness ( $T_{ave}$ ) is zero, but in reality a  $Cr_2Ta$  phase 8 having a thickness ( $T_{ave}$ ) of 5  $\mu m$  or more will be generated.

[0019]

In each of the images showing five locations obtained from EDX analysis, the thickness  $T$  (see FIG. 1) is measured at one randomly-selected location. The thickness ( $T_{ave}$ ) of the  $Cr_2Ta$  phase 8 is calculated as the average value of these thicknesses  $T$ .

[0020]

As described above, a Cr powder is used as a material of the sputtering target material 2. In general, this powder can be manufactured by a grinding method. The mean particle diameter D50 of the Cr powder is preferably 10  $\mu m$  or more and 300  $\mu m$  or less.

[0021]

As described above, a Ta powder is used as a material of the sputtering target material 2. In general, this powder can be manufactured by a chemical reduction method. The mean particle diameter D50 of the Ta powder is preferably 1  $\mu m$  or more and 100  $\mu m$  or less.

[0022]

D50 means a particle diameter at a point where the cumulative volume is 50% in the volume-based cumulative frequency distribution curve determined with the total volume of the powder being 100%. D50 can be determined by a laser diffraction scattering method. Suitable apparatuses for the measurement include a laser diffraction scattering particle size distribution analyzer "Microtrac MT3000" from Nikkiso Co., Ltd. This apparatus analyzes the particle size of powder based on information on the light scattering of the powder that has poured into a cell together with pure water.

[0023]

The Cr powder and the Ta powder are mixed to obtain a

mixed powder. The mixed powder is compacted into the sputtering target material 2. During compaction, the mixed powder is pressurized at high temperatures. A typical pressurization method is hot isostatic pressing (HIP).

- 5 Preferably, the pressure applied during compaction is 50 MPa or more and 300 MPa or less.

[0024]

- Preferably, the temperature during sintering is 1000°C or higher and 1350°C or lower. Pressurization at a temperature of  
 10 1000°C or higher can provide a sputtering target material having a high relative density as the sputtering target material 2. Thus, the temperature is more preferably 1050°C or higher, still more preferably 1100°C or higher. Pressurization at a temperature of 1350°C or lower can result in reduction of Cr<sub>2</sub>Ta  
 15 phase 8 production. Thus, the temperature is more preferably 1300°C or lower, still more preferably 1250°C or lower.

[0025]

- Preferably, the sputtering target material according to the present invention (e.g., sputtering target material 2) has a  
 20 bending strength of 400 MPa or more as measured by a three-point bending test. A target material having a bending strength of 400 MPa or more, when subjected to sputtering, is less likely to crack due to thermal strain. A target material having a bending strength of 400 MPa or more are suitable for  
 25 sputtering with a large amount of electric power. Thus, the bending strength is more preferably 450 MPa or more, particularly preferably 500 MPa or more.

[0026]

- The bending strength is measured according to "JIS Z  
 30 2511." Test pieces are cut out of base materials by wire cutting. The test conditions are as follows:

Size of test pieces: 2 mm (thickness) × 2 mm (width) × 20 mm (length)

Distance between supporting-points: 10 mm

- 35 The load (kN) when the test piece is broken is measured, and then the bending strength BS (MPa) is calculated using the

following formula:

$$BS = (3 \times P \times L) / (2 \times t^2 \times W)$$

t: Thickness of the test piece (mm)

W: Width of the test piece (mm)

5 L: Distance between supporting-points (mm)

P: Load at break (kN)

[0027]

For the sputtering target material according to the present invention (e.g., sputtering target material 2), the ratio  
 10 (Px) of intensity of Cr<sub>2</sub>Ta (220) to intensity of Ta (110) in the X-ray diffraction (XRD) pattern is preferably 10% or less. A sputtering target material with the ratio (Px) being 10% or less shows a high bending strength. A sputtering target material with the ratio (Px) being 10% or less, when subjected to  
 15 sputtering, is less likely to crack due to thermal strain. A sputtering target material with the ratio (Px) being 10% or less is suitable for sputtering with a large amount of electric power. Thus, the ratio (Px) is more preferably 7% or less, still more preferably 5% or less. The ideal ratio (Px) is zero, but in  
 20 reality a sputtering target material will have a ratio (Px) of 1% or more.

[0028]

The size of a test piece to be subjected to XRD is 10 mm × 20 mm × 5 mm. The test piece is cut out of a base material  
 25 by wire cutting. The surface of the test piece is smoothed by polishing. The XRD conditions are as follows:

Radiation source: CuKα

2θ: 20 - 80°

[0029]

30 The measurement employs, for example, a fully automatic multi-purpose X-ray diffraction analyzer "SmartLab SE" from Rigaku Corporation. The diffraction pattern obtained from XRD is used to calculate the ratio (Px).

[0030]

35 The intensity of Ta (110) refers to the intensity of the peak attributed to the (110) plane of Ta among the X-ray

diffraction peaks measured using Cu-K $\alpha$  radiation. The intensity of Cr<sub>2</sub>Ta (220) refers to the intensity of the peak attributed to the (220) plane of Cr<sub>2</sub>Ta among the X-ray diffraction peaks measured using Cu-K $\alpha$  radiation.

5 [0031]

Preferably, the sputtering target material according to the present invention (e.g., sputtering target material 2) has a relative density of 97% or more. A sputtering target material having a relative density of 97% or more is less likely to crack during sputtering. In addition, a sputtering target material having a relative density of 97% or more is less likely to produce particles during sputtering. Thus, the relative density is more preferably 98% or more, still more preferably 99% or more. The ideal relative density is 100%.

15 [0032]

The relative density is a percentage (%) of the true density of a sputtering target material to the theoretical density calculated from the densities of the components of the sputtering target material. The true density (g/cm<sup>3</sup>) is measured by the Archimedes' method. The size of a test piece to be measured is 10 mm × 20 mm × 5 mm. The test piece is cut out of a base material by wire cutting. The surface of the test piece is smoothed by polishing. The true density is calculated by dividing the weight in air of the test piece by the volume of the test piece (= weight in water of the test piece/specific gravity of water at measurement temperature). The theoretical density  $\rho$  (g/cm<sup>3</sup>) is calculated by the following formula:

$$\rho \text{ (g/cm}^3\text{)} = 100 / (C_{\text{Cr}} / \rho_{\text{Cr}} + C_{\text{Ta}} / \rho_{\text{Ta}})$$

30 [0033]

In the formula above,  $C_{\text{Cr}}$  represents the content of Cr (% by mass),  $\rho_{\text{Cr}}$  represents the density of Cr (g/cm<sup>3</sup>),  $C_{\text{Ta}}$  represents the content of Ta (% by mass), and  $\rho_{\text{Ta}}$  represents the density of Ta (g/cm<sup>3</sup>).

35

## EXAMPLES

[0034]

The effects of the present invention will be shown with reference to Examples below, but the present invention should not be construed in a limited manner based on the description  
5 of Examples.

[0035]

[Example 1]

A Cr powder and a Ta powder were prepared. These powders were mixed in a V-type mixer to obtain a mixed powder  
10 with the percentages of the Cr powder being 10 at% and of the Ta powder being 90 at%. The mixed powder was loaded into a can made of carbon steel having a diameter of 200 mm and a length of 10 mm. The powder was degassed under vacuum and then subjected to HIP to prepare a billet. The HIP conditions  
15 were as follows:

Temperature: 1350°C

Pressure: 120 MPa

Holding time: 2 hours.

[0036]

This billet gave a sputtering target material of Example 1.  
20 This sputtering target material is disc-shaped. This sputtering target material had a diameter of 95 mm and a thickness of 2 mm.

[0037]

25 [Examples 2 to 12 and Comparative Examples 1 and 2]

The sputtering target materials of Examples 2 to 12 and Comparative Examples 1 and 2 were obtained in the same manner as in Example 1, except that the compositions and HIP temperatures were as shown in Table 1 below.

30 [0038]

[Comparative Example 3]

The sputtering target material of Comparative Example 3 was obtained by a melting method.

[0039]

35 [Properties of Sputtering target material]

The bending strength, relative density, ratio (Px) of the

intensities in XRD, and thickness (Tave) of the Cr<sub>2</sub>Ta phase of the sputtering target materials were measured by the following methods. The test pieces for measurement were cut out of the billets described above by wire cutting.

5 [0040]

The bending strength was measured according to "JIS Z 2511." The test conditions are as follows:

Size of test pieces: 2 mm (thickness) × 2 mm (width) × 20 mm (length)

10 Distance between supporting-points: 10 mm.

[0041]

The load (kN) when the test piece was broken was measured, and then the bending strength BS (MPa) was calculated using the following formula:

15 
$$BS = (3 \times P \times L) / (2 \times t^2 \times W)$$

t: Thickness of the test piece (mm)

W: Width of the test piece (mm)

L: Distance between supporting-points (mm)

P: Load at break (kN).

20 [0042]

The size of the test piece that was subjected to XRD was 10 mm × 20 mm × 5 mm. The surface of the test piece was smoothed by polishing. The XRD conditions were as follows:

Radiation source: CuKα

25 2θ: 20 - 80°.

[0043]

The measurement employed a fully automatic multi-purpose X-ray diffraction analyzer "SmartLab SE" from Rigaku Corporation.

The intensities of Ta (110) and Cr<sub>2</sub>Ta (220) were determined from

30 the diffraction pattern obtained by XRD, and the ratio (Px) of the intensity of Cr<sub>2</sub>Ta (220) to the intensity of Ta (110) was calculated.

The intensity of Ta (110) refers to the intensity of the peak attributed to the (110) plane of Ta among the X-ray diffraction peaks measured using Cu-Kα radiation. The intensity of Cr<sub>2</sub>Ta

35 (220) refers to the intensity of the peak attributed to the (220) plane of Cr<sub>2</sub>Ta among the X-ray diffraction peaks measured using

Cu-K $\alpha$  radiation.

[0044]

The relative density is a percentage (%) of the true density of a sputtering target material to the theoretical density calculated from the densities of the components of the sputtering target material. The true density (g/cm<sup>3</sup>) was measured by the Archimedes' method. The size of the measured test piece was 10 mm × 20 mm × 5 mm. The surface of the test piece was smoothed by polishing. The true density was calculated by dividing the weight in air of the test piece by the volume of the test piece (= weight in water of the test piece/specific gravity of water at measurement temperature). The theoretical density  $\rho$  (g/cm<sup>3</sup>) was calculated by the following formula:

$$\rho \text{ (g/cm}^3\text{)} = 100 / (C_{\text{Cr}} / \rho_{\text{Cr}} + C_{\text{Ta}} / \rho_{\text{Ta}})$$

[0045]

In the formula above,  $C_{\text{Cr}}$  represents the content of Cr (% by mass),  $\rho_{\text{Cr}}$  represents the density of Cr (g/cm<sup>3</sup>),  $C_{\text{Ta}}$  represents the content of Ta (% by mass), and  $\rho_{\text{Ta}}$  represents the density of Ta (g/cm<sup>3</sup>).

[0046]

Five locations in each of the sputtering target materials were subjected to an energy dispersive X-ray (EDX) analysis. In each of the images showing the five locations obtained from EDX analysis, the thickness  $T$  of the Cr<sub>2</sub>Ta phase (see FIG. 1) was measured at one randomly-selected location. The thicknesses  $T$  were used to calculate the average value, which was considered as the thickness ( $T_{\text{ave}}$ ) of the Cr<sub>2</sub>Ta phase of the sputtering target materials.

[0047]

[Sputtering]

Sputtering was performed using each of the sputtering target materials to form an adhesion layer on a glass substrate. The adhesion layer had a thickness of 20 nm. The sputtering conditions were as follows:

Atmosphere in chamber: After evacuation to  $1 \times 10^{-4}$  Pa

or less, Ar gas with a purity of 99.99% was injected;

Pressure in chamber: 0.6 Pa.

[0048]

- 5 It was visually determined whether the targets after sputtering showed cracking. The results are shown in Table 1 below.

[0049]

[Table 1]

10 Table 1: Results of evaluation of sputtering target materials

|                          | Cr<br>content<br>(at%) | Ta<br>content<br>(at%) | HIP<br>temperature<br>(°C) | Bending<br>strength<br>(MPa) |
|--------------------------|------------------------|------------------------|----------------------------|------------------------------|
| Example 1                | 10                     | 90                     | 1350                       | 420                          |
| Example 2                | 20                     | 80                     | 1300                       | 450                          |
| Example 3                | 30                     | 70                     | 1200                       | 550                          |
| Example 4                | 40                     | 60                     | 1100                       | 520                          |
| Example 5                | 50                     | 50                     | 1050                       | 550                          |
| Example 6                | 50                     | 50                     | 1150                       | 660                          |
| Example 7                | 50                     | 50                     | 1200                       | 640                          |
| Example 8                | 55                     | 45                     | 1300                       | 480                          |
| Example 9                | 60                     | 40                     | 1000                       | 420                          |
| Example 10               | 60                     | 40                     | 1350                       | 420                          |
| Example 11               | 70                     | 30                     | 1100                       | 650                          |
| Example 12               | 80                     | 20                     | 1000                       | 490                          |
| Comparative<br>Example 1 | 40                     | 60                     | 990                        | 370                          |
| Comparative<br>Example 2 | 50                     | 50                     | 1370                       | 310                          |
| Comparative<br>Example 3 | 60                     | 40                     | -                          | 280                          |

Table 1 (continued): Results of evaluation of sputtering target materials

|                       | Relative density (%) | Ratio, Px (%) | Thickness, Tave (μm) | Cracking |
|-----------------------|----------------------|---------------|----------------------|----------|
| Example 1             | 100                  | 9             | 25                   | No       |
| Example 2             | 100                  | 7             | 25                   | No       |
| Example 3             | 100                  | 5             | 20                   | No       |
| Example 4             | 100                  | 4             | 15                   | No       |
| Example 5             | 100                  | 3             | 10                   | No       |
| Example 6             | 100                  | 3             | 10                   | No       |
| Example 7             | 100                  | 3             | 10                   | No       |
| Example 8             | 100                  | 6             | 20                   | No       |
| Example 9             | 97.1                 | 2             | 10                   | No       |
| Example 10            | 100                  | 9             | 30                   | No       |
| Example 11            | 99.5                 | 5             | 20                   | No       |
| Example 12            | 98.2                 | 2             | 15                   | No       |
| Comparative Example 1 | 96.5                 | 2             | 10                   | Yes      |
| Comparative Example 2 | 100                  | 13            | 35                   | Yes      |
| Comparative Example 3 | 100                  | 50            | >50                  | Yes      |

[0050]

- 5           As shown in Table 1, the sputtering target materials in Examples are excellent in strength. These evaluation results demonstrate the advantages of the present invention.

#### INDUSTRIAL AVAILABILITY

10 [0051]

The sputtering target material described above can be used to form a variety of thin films.

## REFERENCE SIGN LIST

[0052]

- |     |                            |
|-----|----------------------------|
| 2   | Sputtering target material |
| 4   | Cr phase                   |
| 5 6 | Ta phase                   |
| 8   | Cr <sub>2</sub> Ta phase   |

CLAIMS

1. A sputtering target material consisting of an alloy,  
wherein the alloy consists of Ta and Cr with the balance being unavoidable impurities, and  
wherein the sputtering target material has a bending strength of 400 MPa or more as measured by a three-point bending test.
2. The sputtering target material according to claim 1, wherein the sputtering target material has a relative density of 97% or more as measured by the Archimedes' method.
3. The sputtering target material according to claim 1 or 2, wherein the sputtering target material has a Cr phase, a Ta phase, and a Cr<sub>2</sub>Ta phase.
4. The sputtering target material according to claim 3, wherein the ratio (Px) of the intensity of Cr<sub>2</sub>Ta (220) to the intensity of Ta (110) in the X-ray diffraction (XRD) pattern is 10% or less.
5. The sputtering target material according to claim 3 or 4, wherein the Cr<sub>2</sub>Ta phase is present on a surface of the Cr phase, and wherein the thickness (Tave) of the Cr<sub>2</sub>Ta phase is 30 μm or less.