

METHOD FOR PREPARING CHROMIUM-SILICON ALLOY SPUTTERING TARGET MATERIAL

TECHNICAL FIELD

5 The present application belongs to the technical field of semiconductors and relates to a method for preparing a sputtering target material, for example, a method for preparing a chromium-silicon alloy sputtering target material.

BACKGROUND

10 Magnetron sputtering is a film coating process in which a target material is bombarded by charged particles so that atoms of the target material overflow from the surface and are uniformly deposited on the substrate. Magnetron sputtering has the advantages of high sputtering rate, low substrate temperature, good adhesion between the substrate and the film, and excellent film uniformity and has been widely used in the manufacturing process of electronic and information industries such as integrated circuits, information storage devices, liquid crystal displays, laser memories, electronic control devices, and the like.

15 The quality of target materials used in magnetron sputtering is one of the key factors affecting the quality of magnetron sputtering coating, and thus, the quality requirements of sputtering target materials are higher than the quality requirements of the conventional material industry. The general quality requirements of sputtering target materials mainly include size, flatness, purity, composition content, density, grain size, and defect control. In addition, sputtering targets
20 have higher or special quality requirements in surface roughness, resistance, grain size uniformity, composition and microstructure uniformity, content and size of oxides, magnetic permeability, ultra-high density, ultra-fine grains, etc.

The silicon-chromium alloy sputtering target material is a novel sputtering target material, and as a good conductor for vacuum sputtering, can be used in the fields of electronic gate materials
25 and electronic films. In order to make the silicon-chromium alloy sputtering target material perform good performance during vacuum sputtering, the silicon-chromium alloy sputtering target material is required to have higher density, higher internal structure uniformity, and better machining conditions.

CN105331939A discloses a silicon-containing alloy target material and a preparation method

thereof, where the preparation method includes the following steps: (1) a prealloyed powder is prepared; (2) cold isostatic pressing is performed on the prealloyed powder to obtain a cold isostatic pressing compact, and the cold isostatic pressing compact is loaded into a can; (3) the can be loaded with the cold isostatic pressing compact is degassed to obtain a degassed ingot blank; (4) hot isostatic pressing is performed on the degassed ingot blank to obtain the pressed ingot blank, and then the can is removed to obtain the pressed blank; and (5) the pressed blank is machined to obtain a silicon-containing alloy target material. However, the cold isostatic pressing requires high pressure and has high requirements on preparation equipment.

CN104419859A discloses a chromium-aluminum-silicon alloy target material. The alloy target material comprises the following components in atom percentage: 5-75 percent of chromium, 10-90 percent of aluminum and 1-20 percent of silicon. The method for preparing the chromium-aluminum-silicon alloy target material includes the following steps: (1) an alloy powder is prepared; (2) cold isostatic pressing is performed on the prepared alloy powder; (3) the cold isostatic pressing-treated blank is degassed; (4) hot isostatic pressing is performed on the degassed blank; and (5) the hot isostatic pressing-treated blank is machined and then cleaned to obtain the desired finished alloy target material. The cold isostatic pressing is still required in the processing process of the chromium-aluminum-silicon alloy target material, which has high requirements on equipment and does not facilitate the reduction of the manufacturing cost of the chromium-aluminum-silicon alloy target material.

CN111058004A discloses a method for preparing a chromium-silicon alloy sputtering target material, where the preparation method includes the following steps: (1) a chromium-silicon alloy powder having a target mass ratio is packed into a mold, and the mold is sealed to perform jolt ramming; (2) the mold after jolt ramming degassing in step (1) is degassed; (3) the degassed mold in step (2) is subjected to hot isostatic pressing at a temperature of 1000°C to 1350°C to obtain a coarse product of the chromium-silicon alloy sputtering target material; and (4) the coarse product of the chromium-silicon alloy sputtering target material is machined to obtain the chromium-silicon alloy sputtering target material. The preparation method can obtain the chromium-silicon alloy sputtering target material through simple treatment of hot isostatic pressing, but the relative density of the target material still needs to be further improved.

Therefore, it is necessary to provide a new method for preparing a chromium-silicon alloy sputtering target material so that the sputtering target prepared by this preparation method has higher density and denser internal structure.

SUMMARY

The object of the present application is to provide a method for preparing a chromium-silicon alloy sputtering target material. Through the preparation method, the prepared chromium-silicon alloy sputtering target material is high in relative density, has a uniformly dense microcosmic structure, and is free of voids and layered structures, which can provide good performance guarantee for subsequent sputtering use.

To achieve this object, the present application adopts the following technical solutions.

The present application provides a method for preparing a chromium-silicon alloy sputtering target material. The preparation method includes the following steps:

(1) mixing a chromium powder and a silicon powder under a non-oxidizing atmosphere to obtain a mixed powder;

(2) performing die filling, and performing vacuum hot-pressing sintering on the mixed powder obtained after the die filling; and

(3) performing furnace cooling after the vacuum hot-pressing sintering is finished to obtain the chromium-silicon alloy sputtering target material.

Optionally, the non-oxidizing atmosphere in step (1) includes a nitrogen atmosphere and/or an inert atmosphere.

Optionally, the inert atmosphere includes any one or a combination of at least two of a helium atmosphere, a neon atmosphere, an argon atmosphere or a krypton atmosphere.

In the present application, the gas used in the inert atmosphere includes any one or a combination of at least two of helium, neon, argon or krypton, and typical but non-limiting combinations include a combination of helium and neon, a combination of neon and argon, a combination of argon and krypton, a combination of neon and krypton, a combination of helium and argon, a combination of argon, neon and helium or a combination of helium, neon, argon and krypton.

Optionally, the chromium powder has a purity of greater than or equal to 99.95%, for example, 99.95%, 99.96%, 99.97%, 99.98%, 99.99%, 99.992%, 99.994%, 99.995%, 99.996%, 99.998%,

or 99.999%. However, the purity is not limited to the values listed above, and other unlisted values within this value range are also applicable.

5 Optionally, the chromium powder has a particle size of less than 45 μm , for example, 5 μm , 10 μm , 15 μm , 20 μm , 25 μm , 30 μm , 35 μm , 40 μm , or 44 μm . However, the particle size is not limited to the values listed above, and other unlisted values within this value range are also applicable.

10 Optionally, the silicon powder has a purity of greater than or equal to 99.99%, for example, 99.99%, 99.992%, 99.994%, 99.995%, 99.996%, 99.998%, or 99.999%. However, the purity is not limited to the values listed above, and other unlisted values within this value range are also applicable.

Optionally, the silicon powder has a particle size of less than 3 μm , for example, 0.5 μm , 1 μm , 1.5 μm , 2 μm , 2.5 μm , 2.7 μm , or 2.8 μm . However, the particle size is not limited to the values listed above, and other unlisted values within this value range are also applicable.

15 Optionally, the mass percentage of silicon in the mixed powder is 50 wt% to 70 wt%, for example, 50 wt%, 55 wt%, 60 wt%, 65 wt%, or 70 wt%. However, the mass percentage is not limited to the values listed above, and other unlisted values within this value range are also applicable.

Optionally, the mixing in step (1) is performed in a ball mill.

20 Optionally, the mill balls when the mixing is performed in the ball mill are silicon balls and/or chromium balls.

Optionally, a ratio of material to grinding media when the mixing is performed in the ball mill is (8 to 12):1, for example, 8:1, 9:1, 10:1, 11:1, or 12:1. However, the ratio of material to grinding media is not limited to the values listed above, and other unlisted values within this value range are also applicable.

25 Optionally, the mixing in step (1) is performed for at least 24 hours, for example, 24 hours, 25 hours, 26 hours, 27 hours, 28 hours, or 30 hours. However, the time is not limited to the values listed above, and other unlisted values within this value range are also applicable.

Optionally, the die filling in step (2) is to feed the mixed powder into a graphite mold.

Optionally, the flatness after the die filling in step (2) is less than or equal to 0.5 mm, for example, 0.1 mm, 0.2 mm, 0.3 mm, 0.4 mm, or 0.5 mm. However, the flatness is not limited to the values listed above, and other unlisted values within this value range are also applicable.

5 In the present application, the vacuum hot-pressing sintering is performed in a hot pressure sintering furnace.

Optionally, the vacuum hot-pressing sintering in step (2) includes the following steps:

(a) vacuumizing to an absolute vacuum degree of below 100 Pa, and performing first programmed temperature rise, where the first programmed temperature rise is performed at a pressure which does not exceed 5 MPa;

10 (b) performing first heat preservation after the first programmed temperature rise is finished, and sequentially performing second programmed temperature rise and second heat preservation; and

(c) performing pressurization after the second heat preservation is finished, and performing heat preservation and pressure maintaining after the pressurization is finished.

15 Optionally, the first programmed temperature rise in step (a) is performed at a heating rate of 6°C/minute to 10°C/minute, for example, 6°C/minute, 7°C/minute, 8°C/minute, 9°C/minute, or 10°C/minute. However, the heating rate is not limited to the values listed above, and other unlisted values within this value range are also applicable.

20 Optionally, the first programmed temperature rise in step (a) ends at a temperature of 950°C to 1050°C, for example, 950°C, 960°C, 970°C, 980°C, 990°C, 1000°C, 1010°C, 1020°C, 1030°C, 1040°C, or 1050°C. However, the temperature is not limited to the values listed above, and other unlisted values within this value range are also applicable.

25 Optionally, the first heat preservation in step (b) is performed for 50 minutes to 80 minutes, for example, 50 minutes, 55 minutes, 60 minutes, 65 minutes, 70 minutes, 75 minutes, or 80 minutes. However, the time is not limited to the listed values, and other unlisted values within this value range are also applicable.

Optionally, the second programmed temperature rise in step (b) is performed at a heating rate of 3°C/minute to 6°C/minute, for example, 3°C/minute, 3.5°C/minute, 4°C/minute, 4.5°C/minute,

5°C/minute, 5.5°C/minute, or 6°C/minute. However, the heating rate is not limited to the values listed above, and other unlisted values within this value range are also applicable.

Optionally, the heating rate of the second programmed temperature rise is less than the heating rate of the first programmed temperature rise.

- 5 Optionally, the second programmed temperature rise in step (b) ends at a temperature of 1100°C to 1300°C, for example, 1100°C, 1110°C, 1120°C, 1130°C, 1140°C, 1150°C, 1160°C, 1170°C, 1180°C, 1190°C, 1200°C, 1220°C, 1240°C, 1250°C, 1270°C, 1280°C, or 1300°C. However, the temperature is not limited to the values listed above, and other unlisted values within this value range are also applicable.
- 10 Optionally, the second heat preservation in step (b) is performed for at least 1 hour, for example, 1 hour, 1.5 hours, 2 hours, 2.5 hours, 3 hours, 4 hours, or 5 hours. However, the time is not limited to the values listed above, and other unlisted values within this value range are also applicable.

- 15 Optionally, the pressurization in step (c) is to perform uniform pressurization to 30 MPa within 2 hours, where the pressurization is performed at a constant temperature.

Optionally, the heat preservation and pressure maintaining in step (c) are performed for 80 minutes to 100minutes, for example, 80 minutes, 85 minutes, 90 minutes, 95 minutes, or 100 minutes. However, the time is not limited to the listed values, and other unlisted values within this value range are also applicable.

- 20 As an optional technical solution of the preparation method of the present application, the preparation method includes the following steps:

- (1) mixing a chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm under a non-oxidizing atmosphere to obtain a mixed powder; where the mass percentage of silicon in the mixed powder is 50 wt% to 70 wt%;
- 25 (2) performing die filling in a graphite mold until the flatness is less than or equal to 0.5 mm, and performing vacuum hot pressing sintering on the mixed powder obtained after the die filling; and
- (3) performing furnace cooling after the vacuum hot-pressing sintering is finished to obtain the

chromium-silicon alloy sputtering target material.

The vacuum hot-pressing sintering in step (2) includes the following steps:

- 5 (a) vacuumizing to an absolute vacuum degree of below 100 Pa, and performing first programmed temperature rise, where the first programmed temperature rise is performed at a pressure which does not exceed 5 MPa, and the first programmed temperature rise is performed at a heating rate of 6°C/minute to 10°C/minute to 950°C to 1050°C;
- 10 (b) performing heat preservation for 50 minutes to 80 minutes after the first programmed temperature rise is finished, and sequentially performing second programmed temperature rise and second heat preservation; where the second programmed temperature rise is performed at a heating rate of 3°C/minute to 6°C/minute to 1100°C to 1300°C, and the second heat preservation is performed for at least 1 hour; and
- (c) performing uniform pressurization to 30 MPa within 2 hours at a constant temperature after the second heat preservation is finished, and performing heat preservation and pressure maintaining for 80 minutes to 100 minutes after the pressurization is finished.
- 15 Any numerical range described in the present application not only includes the above-exemplified point values, but also includes any point values within the numerical range which are not exemplified. For the sake of length and simplicity, specific point values included in the range are not exhaustively listed in the present application.

Compared with the existing art, the present application has the following beneficial effects.

- 20 The preparation method provided in the present application is particularly suitable for the chromium-silicon alloy sputtering target material with a high silicon content by adjusting the mixing method of the chromium powder and the silicon powder and the specific operation of the vacuum hot-pressing sintering, and the prepared chromium-silicon alloy sputtering target material has a high density of greater than or equal to 99% and a uniformly dense microcosmic structure and is free of voids and layered structures, which can provide good performance
- 25 guarantee for subsequent sputtering use.

DETAILED DESCRIPTION

The technical solutions of the present application are further described below through the

detailed description.

Example 1

This example provides a method for preparing a chromium-silicon alloy sputtering target material. The preparation method includes the following steps.

5 (1) A chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm were mixed under a nitrogen atmosphere to obtain a mixed powder; where the mass percentage of silicon in the mixed powder is 60 wt%, the purity of the chromium powder was greater than or equal to 99.95 wt%, the purity of the silicon powder was greater than or equal to 99.99 wt%, and the mixing was performed in a ball mill with silicon
10 balls and at the ratio of material to grinding media of 10:1 for 24 hours.

(2) Die filling was performed in a graphite mold until the flatness was less than or equal to 0.5 mm, and vacuum hot pressing sintering was performed on the mixed powder obtained after the die filling.

(3) Natural cooling was performed after the vacuum hot-pressing sintering was finished to
15 obtain the chromium-silicon alloy sputtering target material.

The vacuum hot-pressing sintering in step (2) includes the following steps.

(a) Vacuumizing was performed until the absolute vacuum degree was below 100 Pa, and first programmed temperature rise was performed; where the first programmed temperature rise was performed at a pressure that did not exceed 5 MPa, and the first programmed temperature rise
20 was performed at a heating rate of 8°C/minute to 1000°C.

(b) Heat preservation was performed for 65 minutes after the first programmed temperature rise was finished, and second programmed temperature rise and second heat preservation were sequentially performed; where the second programmed temperature rise was performed at a heating rate of 4°C/minute to 1150°C, and the second heat preservation was performed for 1
25 hour.

(c) Uniform pressurization was performed to 30 MPa within 2 hours at a constant temperature after the second heat preservation was finished, and heat preservation and pressure maintaining were performed for 90 minutes after the pressurization was finished.

Example 2

This example provides a method for preparing a chromium-silicon alloy sputtering target material. The preparation method includes the following steps.

- 5 (1) A chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm were mixed under a helium atmosphere to obtain a mixed powder; where the mass percentage of silicon in the mixed powder is 55 wt%, the purity of the chromium powder was greater than or equal to 99.95 wt%, the purity of the silicon powder was greater than or equal to 99.99 wt%, and the mixing was performed in a ball mill with silicon balls and at the ratio of material to grinding media of 9:1 for 24 hours.
- 10 (2) Die filling was performed in a graphite mold until the flatness was less than or equal to 0.5 mm, and vacuum hot pressing sintering was performed on the mixed powder obtained after the die filling.
- (3) Natural cooling was performed after the vacuum hot-pressing sintering was finished to obtain the chromium-silicon alloy sputtering target material.
- 15 The vacuum hot-pressing sintering in step (2) includes the following steps.
 - (a) Vacuumizing was performed until the absolute vacuum degree was below 100 Pa, and first programmed temperature rise was performed; where the first programmed temperature rise was performed at a pressure that did not exceed 5 MPa, and the first programmed temperature rise was performed at a heating rate of 7°C/minute to 980°C.
 - 20 (b) Heat preservation was performed for 70 minutes after the first programmed temperature rise was finished, and second programmed temperature rise and second heat preservation were sequentially performed; where the second programmed temperature rise was performed at a heating rate of 4°C/minute to 1120°C, and the second heat preservation was performed for 1 hour.
 - 25 (c) Uniform pressurization was performed to 30 MPa within 2 hours at a constant temperature after the second heat preservation was finished, and heat preservation and pressure maintaining were performed for 85 minutes after the pressurization was finished.

Example 3

This example provides a method for preparing a chromium-silicon alloy sputtering target material. The preparation method includes the following steps.

- 5 (1) A chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm were mixed under a neon atmosphere to obtain a mixed powder; where the mass percentage of silicon in the mixed powder is 65 wt%, the purity of the chromium powder was greater than or equal to 99.95 wt%, the purity of the silicon powder was greater than or equal to 99.99 wt%, and the mixing was performed in a ball mill with silicon balls and at the ratio of material to grinding media of 11:1 for 24 hours.
- 10 (2) Die filling was performed in a graphite mold until the flatness was less than or equal to 0.5 mm, and vacuum hot pressing sintering was performed on the mixed powder obtained after the die filling.
- (3) Natural cooling was performed after the vacuum hot-pressing sintering was finished to obtain the chromium-silicon alloy sputtering target material.

The vacuum hot-pressing sintering in step (2) includes the following steps.

- 15 (a) Vacuumizing was performed until the absolute vacuum degree was below 100 Pa, and first programmed temperature rise was performed; where the first programmed temperature rise was performed at a pressure that did not exceed 5 MPa, and the first programmed temperature rise was performed at a heating rate of 9°C/minute to 1020°C.
- 20 (b) Heat preservation was performed for 60 minutes after the first programmed temperature rise was finished, and second programmed temperature rise and second heat preservation were sequentially performed; where the second programmed temperature rise was performed at a heating rate of 5°C/minute to 1180°C, and the second heat preservation was performed for 1 hour.
- 25 (c) Uniform pressurization was performed to 30 MPa within 2 hours at a constant temperature after the second heat preservation was finished, and heat preservation and pressure maintaining were performed for 95 minutes after the pressurization was finished.

Example 4

This example provides a method for preparing a chromium-silicon alloy sputtering target

material. The preparation method includes the following steps.

(1) A chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm were mixed under an argon atmosphere to obtain a mixed powder; where the mass percentage of silicon in the mixed powder is 50 wt%, the purity of the chromium powder was greater than or equal to 99.95 wt%, the purity of the silicon powder was greater than or equal to 99.99 wt%, and the mixing was performed in a ball mill with chromium balls and at the ratio of material to grinding media of 8:1 for 24 hours.

(2) Die filling was performed in a graphite mold until the flatness was less than or equal to 0.5 mm, and vacuum hot pressing sintering was performed on the mixed powder obtained after the die filling.

(3) Natural cooling was performed after the vacuum hot-pressing sintering was finished to obtain the chromium-silicon alloy sputtering target material.

The vacuum hot-pressing sintering in step (2) includes the following steps.

(a) Vacuumizing was performed until the absolute vacuum degree was below 100 Pa, and first programmed temperature rise was performed; where the first programmed temperature rise was performed at a pressure that did not exceed 5 MPa, and the first programmed temperature rise was performed at a heating rate of 6°C/minute to 950°C.

(b) Heat preservation was performed for 80 minutes after the first programmed temperature rise was finished, and second programmed temperature rise and second heat preservation were sequentially performed; where the second programmed temperature rise was performed at a heating rate of 3°C/minute to 1100°C, and the second heat preservation was performed for 1 hour.

(c) Uniform pressurization was performed to 30 MPa within 2 hours at a constant temperature after the second heat preservation was finished, and heat preservation and pressure maintaining were performed for 100 minutes after the pressurization was finished.

Example 5

This example provides a method for preparing a chromium-silicon alloy sputtering target material. The preparation method includes the following steps.

(1) A chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm were mixed under a krypton atmosphere to obtain a mixed powder; where the mass percentage of silicon in the mixed powder is 70 wt%, the purity of the chromium powder was greater than or equal to 99.95 wt%, the purity of the silicon powder was greater than or equal to 99.99 wt%, and the mixing was performed in a ball mill with chromium balls and at the ratio of material to grinding media of 12:1 for 24 hours.

(2) Die filling was performed in a graphite mold until the flatness was less than or equal to 0.5 mm, and vacuum hot pressing sintering was performed on the mixed powder obtained after the die filling.

(3) Natural cooling was performed after the vacuum hot-pressing sintering was finished to obtain the chromium-silicon alloy sputtering target material.

The vacuum hot-pressing sintering in step (2) includes the following steps.

(a) Vacuumizing was performed until the absolute vacuum degree was below 100 Pa, and first programmed temperature rise was performed; where the first programmed temperature rise was performed at a pressure that did not exceed 5 MPa, and the first programmed temperature rise was performed at a heating rate of 10°C/minute to 1050°C.

(b) Heat preservation was performed for 50 minutes after the first programmed temperature rise was finished, and second programmed temperature rise and second heat preservation were sequentially performed; where the second programmed temperature rise was performed at a heating rate of 6°C/minute to 1300°C, and the second heat preservation was performed for 1 hour.

(c) Uniform pressurization was performed to 30 MPa within 2 hours at a constant temperature after the second heat preservation was finished, and heat preservation and pressure maintaining were performed for 80 minutes after the pressurization was finished.

Example 6

This example provides a method for preparing a chromium-silicon alloy sputtering target material, which is the same as the preparation method in Example 1 except that the mixing in step (1) was performed without any mill ball.

Example 7

This example provides a method for preparing a chromium-silicon alloy sputtering target material, which is the same as the preparation method in Example 1 except that the mixing in step (1) was performed for 20 hours.

5 Example 8

This example provides a method for preparing a chromium-silicon alloy sputtering target material, which is the same as the preparation method in Example 1 except that the first programmed temperature rise in step (a) was performed at a pressure of 6 MPa to 10 MPa.

Example 9

- 10 This example provides a method for preparing a chromium-silicon alloy sputtering target material, which is the same as the preparation method in Example 1 except that the first programmed temperature rise in step (a) was performed at a heating rate of 12°C/minute.

Example 10

- 15 This example provides a method for preparing a chromium-silicon alloy sputtering target material, which is the same as the preparation method in Example 1 except that the second programmed temperature rise in step (b) was performed at a heating rate of 8°C/minute.

Example 11

This example provides a method for preparing a chromium-silicon alloy sputtering target material. The preparation method includes the following steps.

- 20 (1) A chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm were mixed under a nitrogen atmosphere to obtain a mixed powder; where the mass percentage of silicon in the mixed powder is 60 wt%, the purity of the chromium powder was greater than or equal to 99.95 wt%, the purity of the silicon powder was greater than or equal to 99.99 wt%, and the mixing was performed in a ball mill with silicon
25 balls and at the ratio of material to grinding media of 10:1 for 24 hours.

(2) Die filling was performed in a graphite mold until the flatness was less than or equal to 0.5

mm, and vacuum hot pressing sintering was performed on the mixed powder obtained after the die filling.

(3) Furnace cooling was performed after the vacuum hot-pressing sintering was finished to obtain the chromium-silicon alloy sputtering target material.

5 The vacuum hot-pressing sintering in step (2) includes the following steps.

10 (I) Vacuumizing was performed until the absolute vacuum degree was below 100 Pa, first programmed temperature rise was performed, where the first programmed temperature rise was performed at a pressure that did not exceed 5 MPa, and the first programmed temperature rise was performed at a heating rate of 8°C/minute to 1150°C, and then heat preservation was performed for 1 hour.

(II) Uniform pressurization was performed to 30 MPa within 2 hours at a constant temperature after the heat preservation was finished, and heat preservation and pressure maintaining were performed for 90 minutes after the pressurization was finished.

15 The relative density and internal structure uniformity of the chromium-silicon alloy sputtering target materials provided in Examples 1 to 11 were tested, and the test results are shown in Table 1.

Table 1

	Relative Density/%	Internal structure uniformity
Example 1	99.9	uniformly dense at the microcosmic level without voids and layered structures
Example 2	99.7	uniformly dense at the microcosmic level without voids and layered structures
Example 3	99.8	uniformly dense at the microcosmic level without voids and layered structures
Example 4	99.6	uniformly dense at the microcosmic level without voids and layered structures
Example 5	99.7	uniformly dense at the microcosmic level without voids and layered structures
Example 6	97.8	nonuniform in the microcosmic structure with the existence of voids
Example 7	95.4	nonuniform in the microcosmic structure with the existence of voids

Example 8	87.9	nonuniform in the microcosmic structure of the resulting target material with the existence of cracking phenomena
Example 9	86.3	nonuniform in the microcosmic structure of the resulting target material with the existence of cracking phenomena
Example 10	87.0	nonuniform in the microcosmic structure of the resulting target material with the existence of cracking phenomena
Example 11	91.6	nonuniform in the microcosmic structure of the resulting target material with the existence of cracking phenomena

As can be seen from Table 1, the chromium-silicon alloy sputtering target material prepared by the preparation method of the present application had a relative density of greater than or equal to 99% and a uniformly dense microscopic structure, without voids and layered structures, which can improve good performance guarantee for subsequent sputtering use.

- 5 To sum up, the preparation method provided in the present application is particularly suitable for the chromium-silicon alloy sputtering target material with a high silicon content by adjusting the mixing method of the chromium powder and the silicon powder and the specific operation of the vacuum hot-pressing sintering, and the prepared chromium-silicon alloy sputtering target material has a high relative density of greater than or equal to 99% and a uniformly dense
- 10 microcosmic structure and is free of voids and layered structures, which can provide good performance guarantee for subsequent sputtering use.

Objects, technical solutions and beneficial effects of the present application are further described in detail through the preceding specific examples, and it should be understood that the examples described above are just specific examples of the present application and not to limit

15 the present application.

What is claimed is:

1. A method for preparing a chromium-silicon alloy sputtering target material, comprising the following steps:

5 (1) mixing a chromium powder and a silicon powder under a non-oxidizing atmosphere to obtain a mixed powder;

(2) performing die filling, and performing vacuum hot-pressing sintering on the mixed powder obtained after the die filling; and

(3) performing furnace cooling after the vacuum hot-pressing sintering is finished to obtain the chromium-silicon alloy sputtering target material.

10 2. The preparation method according to claim 1, wherein the chromium powder has a purity of greater than or equal to 99.95%.

3. The preparation method according to claim 1 or 2, wherein the mass percentage of silicon in the mixed powder is 50 wt% to 70 wt%.

15 4. The preparation method according to any one of claims 1 to 3, wherein the chromium powder has a particle size of less than 45 μm ;

optionally, the silicon powder has a purity of greater than or equal to 99.99%; and

optionally, the silicon powder has a particle size of less than 3 μm .

5. The preparation method according to any one of claims 1 to 4, wherein the non-oxidizing atmosphere in step (1) comprises a nitrogen atmosphere and/or an inert atmosphere; and

20 optionally, the inert atmosphere comprises any one or a combination of at least two of a helium atmosphere, a neon atmosphere, an argon atmosphere or a krypton atmosphere.

6. The preparation method according to any one of claims 1 to 5, wherein the mixing in step (1) is performed in a ball mill;

25 optionally, the mill balls when the mixing is performed in the ball mill are silicon balls and/or chromium balls;

optionally, a ratio of material to grinding media when the mixing is performed in the ball mill is (8 to 12):1; and

optionally, the mixing in step (1) is performed for at least 24 hours.

7. The preparation method according to any one of claims 1 to 6, wherein the die filling in step (2) is to feed the mixed powder into a graphite mold; and

optionally, the flatness after the die filling in step (2) is less than or equal to 0.5 mm.

5 8. The preparation method according to any one of claims 1 to 7, wherein the vacuum hot-pressing sintering comprises the following steps:

(a) vacuumizing to an absolute vacuum degree of below 100 Pa, and performing first programmed temperature rise, wherein the first programmed temperature rise is performed at a pressure which does not exceed 5 MPa;

10 (b) performing first heat preservation after the first programmed temperature rise is finished, and sequentially performing second programmed temperature rise and second heat preservation; and

(c) performing pressurization after the second heat preservation is finished, and performing heat preservation and pressure maintaining after the pressurization is finished.

15 9. The preparation method according to claim 8, wherein the first programmed temperature rise in step (a) is performed at a heating rate of 6°C/minute to 10°C/minute; and

optionally, the first programmed temperature rise in step (a) ends at a temperature of 950°C to 1050°C.

20 10. The preparation method according to claim 8 or 9, wherein the first heat preservation in step (b) is performed for 50 minutes to 80 minutes;

optionally, the second programmed temperature rise in step (b) is performed at a heating rate of 3°C/minute to 6°C/minute;

optionally, the second programmed temperature rise in step (b) ends at a temperature of 1100°C to 1300°C; and

25 optionally, the second heat preservation in step (b) is performed for at least 1 hour.

11. The preparation method according to any one of claims 8 to 10, wherein the pressurization in step (c) is to perform uniform pressurization to 30 MPa within 2 hours, wherein the pressurization is performed at a constant temperature; and

optionally, the heat preservation and pressure maintaining in step (c) are performed for 80 minutes to 100minutes.

12. The preparation method according to any one of claims 1 to 11, comprising the following steps:

- 5 (1) mixing a chromium powder with a particle size of less than 45 μm and a silicon powder with a particle size of less than 3 μm under a non-oxidizing atmosphere to obtain a mixed powder; wherein the mass percentage of silicon in the mixed powder is 50 wt% to 70 wt%;
- (2) performing die filling in a graphite mold until the flatness is less than or equal to 0.5 mm, and performing vacuum hot-pressing sintering on the mixed powder obtained after the die
10 filling; and
- (3) performing furnace cooling after the vacuum hot-pressing sintering is finished to obtain the chromium-silicon alloy sputtering target material;

wherein the vacuum hot-pressing sintering in step (2) comprises the following steps:

- 15 (a) vacuumizing to an absolute vacuum degree of below 100 Pa, and performing first programmed temperature rise, where the first programmed temperature rise is performed at a pressure which does not exceed 5 MPa, and the first programmed temperature rise is performed at a heating rate of 6°C/minute to 10°C/minute to 950°C to 1050°C;
- (b) performing heat preservation for 50 minutes to 80 minutes after the first programmed temperature rise is finished, and sequentially performing second programmed temperature rise and second heat preservation; wherein the second programmed temperature rise is performed at
20 a heating rate of 3°C/minute to 6°C/minute to 1100°C to 1300°C, and the second heat preservation is performed for at least 1 hour; and
- (c) performing uniform pressurization to 30 MPa within 2 hours at a constant temperature after the second heat preservation is finished, and performing heat preservation and pressure
25 maintaining for 80 minutes to 100 minutes after the pressurization is finished.