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A preparation method of molybdenum target material

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ABSTRACT

This invention relates to a preparation method of molybdenum target material, pertaining to the technical field of sputtering target material preparation. The preparation method of molybdenum target material of the invention comprises the following steps: first, sinter molybdenum materials into molybdenum ingots; then, purify by electron beam melting; finally, obtain molybdenum target material by circumferential rolling and vacuum annealing. The method solves problems of high impurity content and uneven texture distribution of molybdenum target materials. Molybdenum target materials prepared by this method have higher purity and intensity, more even texture distribution. They can form specific crystal orientation and acquire better sputtering performance.

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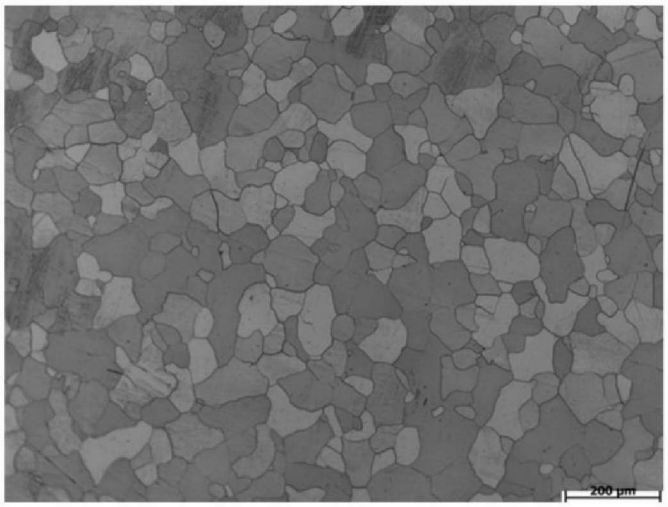

Figure 1

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AUSTRALIA

PATENTS ACT 1990

PATENT SPECIFICATION

FOR THE INVENTION ENTITLED:-

A preparation method of molybdenum target material

The invention is described in the following statement:-

A preparation method of molybdenum target material

TECHNICAL FIELD

[0001] This invention relates to a preparation method of molybdenum target material, pertaining to the technical field of sputtering target material preparation.

BACKGROUND

[0002] Molybdenum and molybdenum alloy have excellent properties such as good electrical and thermal conductivity, low thermal expansion coefficient, good corrosion resistance and environmental friendliness. The specific impedance and film stress of molybdenum film are only half of those of chromium. Therefore, molybdenum/molybdenum alloy target materials have been widely used in electronic components and electronic products, such as thin film semiconductor tube-liquid crystal display (TFT-LCD), plasma display, field emission display and touch screen, and can also be used in fields such as back electrode of solar cells and glass coating. Target materials require high purity, density and specific texture. Molybdenum target materials prepared by traditional powder metallurgy method have the disadvantages of high impurity content and low density. Rolling is a post-processing technology commonly used for producing molybdenum plates and sputtering target materials. Molybdenum plate rolled by traditional method is uneven in texture distribution in the thickness direction of plate, and the existence of texture gradient may affect the sputtering performance of molybdenum sputtering target materials. The

Chinese invention patent application of Appl. No. CN103567445A discloses a preparation method of molybdenum target material. Its technical proposal is: densify molybdenum powder by adopting processes of static pressing, isostatic cold pressing and induction sintering; then obtain molybdenum target material by hot rolling and annealing treatment. Molybdenum target materials obtained by this method has a purity not lower than 99. 95%, which is, however, not high enough.

SUMMARY

[0003] To solve the above problem, this invention aims to provide a preparation method of molybdenum target material, by which the molybdenum target materials prepared has low impurity content and even texture distribution.

[0004] To realize the above objective, the technical proposal adopted by this invention is:

[0005] A preparation method of molybdenum target material, comprising the following steps:

[0006] (1) Roll and sinter molybdenum materials to form cylinder molybdenum ingots;

[0007] (2) Prepare molybdenum target ingots with cylinder molybdenum ingots from step (1) by circumferential rolling;

[0008] (3) Acquire molybdenum target materials with molybdenum target ingots from step (2) by vacuum annealing.

[0009] Molybdenum target material prepared by the above method has a purity ≥99 .99%, density up to 99% and better sputtering performance.

[0010] The said molybdenum material in step (1) is molybdenum powder with granularity being 2～3μm. The molybdenum material has a uniform particle size, better dispersion of particles, and more uniform distribution of molybdenum material during rolling.

[0011] The said molybdenum material in step (1) has a purity ≥99 .95 %. The higher the purity of molybdenum material, the better the comprehensive performance of the molybdenum target material.

[0012] The said sintering temperature in step (1) is 1800～1850℃ and duration is 6～8h. Under these conditions, the green molybdenum plate ingots can be sintered into molybdenum plate ingots, of which the density can reach up to 9 .6～9 .9g/cm^3.

[0013] The said cylinder molybdenum ingots in step (1) are made into purified cylinder molybdenum ingots by purification with a purity ≥99 .99%. Molybdenum target material made from molybdenum ingots with a purity ≥99 .99% can improve and control the organizational structure of material, endowing more excellent performance of molybdenum target material.

[0014] For the said circumferential rolling in step (2), the rolling direction of a consequent pass forms an included angle of 120 °～150 ° with that of the previous pass. This rolling method can improve problems of concentrated orientation and texture gradient of molybdenum target material; improve sputtering performance of molybdenum target material.

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[0015] The said molybdenum target ingots in step (2) have a total deformation of 70～90%. Molybdenum target material with a total deformation of molybdenum target ingots of 70～90% has more uniform and fine particles on the surface, as well as more obvious crystal particle orientation.

[0016] The said vacuum annealing temperature in step (3) is 1050～1150℃ and the duration is 1～2h. Molybdenum target materials prepared under this condition have more fine and uniform crystal particles with granularity reaching up to 50～100μm.

[0017] The vacuum degree of the said vacuum annealing in step (3) is at least 1 .5×10^{-3} Pa. Under this vacuum condition, the oxidation volatility of molybdenum can be reduced to obtain better stability and safer operation environment.

BRIEF DESCRIPTION OF THE FIGURES

[0018] Figure 1 is a microstructure diagram of molybdenum target material in test case of the invention.

DESCRIPTION OF THE INVENTION

[0019] Specific embodiments of the present invention are further explained below with reference to the accompanying drawings.

[0020] In specific embodiments of the present invention, the content of K and O in molybdenum material are not higher than 20ppm and 500ppm,

and the content of impurities C, N and O in purified molybdenum ingots are all lower than 5ppm.

[0021] Embodiment 1 for the preparation method of molybdenum target material

[0022] Preparation method of molybdenum target material for the present embodiment, comprising the following steps:

[0023] (1) Fill in molybdenum powder with purity being 99.95% and granularity being 2μm, with uniform particle size in normal distribution and good dispersibility, without obvious agglomeration; apply isostatic cold pressing under a pressure of 180MPa for 15min to get green molybdenum plate ingots, which are then subjected to induction sintering in hydrogen atmosphere at medium frequency for 8h at a sintering temperature of 1800℃ to finally obtain cylinder molybdenum ingots;

[0024] (2) Purify the sintered molybdenum ingots from step (1) by electron beam melting to get purified molybdenum ingots with a purity of 99.99%;

[0025] (3) Forge and cog down the purified molybdenum ingots from step (2), and then subject them to circumferential rolling to get molybdenum target ingots, of which the total deformation is 75%. The circumferential rolling is carried out in a manner of continuously changing the rolling direction, and the rolling direction of a consequent pass forms an angle of 135° with that of the previous pass; each eight passes is one cycle to obtain molybdenum target ingots;

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[0026] (4) Subject the molybdenum target ingots from step (3) in a vacuum annealing furnace to anneal at a temperature of 1050℃ and vacuum degree of 1 .5×10^{-3} Pa for 1 h; finally, process according to specified specifications to prepare the molybdenum target material.

[0027] Molybdenum target materials obtained by this method has a purity of 99.99% and density of 99%, with contents of C, N and O less than 5ppm, with uniform particle size of molybdenum target material, mean crystal particle diameter of 60μm and crystallization orientation taking {100}<011> as the preferred texture orientation.

[0028] Embodiment 2 for the preparation method of molybdenum target material.

[0029] Preparation method of molybdenum target material for the present embodiment, comprising the following steps:

[0030] (1) Fill in molybdenum powder with purity being 99.97% and granularity being 2μm, with uniform particle size in normal distribution and good dispersibility, without obvious agglomeration; apply isostatic cold pressing under a pressure of 185MPa for 13min to get green molybdenum plate ingots, which are then subjected to induction sintering in hydrogen atmosphere at medium frequency for 7h at a sintering temperature of 1820℃ to finally obtain cylinder molybdenum ingots;

[0031] (2) Purify the sintered molybdenum ingots from step (1) by electron beam melting to get purified molybdenum ingots with a purity of 99.996%;

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[0032] (3) Forge and cog down the purified molybdenum ingots from step (2), and then subject them to circumferential rolling to get molybdenum target ingots, of which the total deformation is 70%. The circumferential rolling is carried out in a manner of continuously changing the rolling direction, and the rolling direction of a consequent pass forms an angle of 120° with that of the previous pass; each 6 passes is one cycle to obtain molybdenum target ingots;

[0033] (4) Subject the molybdenum target ingots from step (3) in a vacuum annealing furnace to anneal at a temperature of 1100℃ and vacuum degree of 1 .5×10^{-3} Pa for 1h; finally, process according to specified specifications to prepare the molybdenum target material.

[0034] Molybdenum target materials obtained by this method has a purity of 99.996% and density of 99%, with contents of C, N and O less than 5ppm, with uniform particle size of molybdenum target material, mean crystal particle diameter of 60μm and crystallization orientation taking {100}<011> as the preferred texture orientation.

[0035] Embodiment 3 for the preparation method of molybdenum target material

[0036] Preparation method of molybdenum target material for the present embodiment, comprising the following steps:

[0037] (1) Fill in molybdenum powder with purity being 99.99% and granularity being 2μm, with uniform particle size in normal distribution and good dispersibility, without obvious agglomeration; apply isostatic cold pressing under a pressure of 200MPa for 10min to get green molybdenum plate ingots, which are then subjected to induction sintering in hydrogen

atmosphere at medium frequency for 6h at a sintering temperature of 1850℃ to finally obtain cylinder molybdenum ingots;

[0038] (2) Purify the sintered molybdenum ingots from step (1) by electron beam melting to get purified molybdenum ingots with a purity of 99.999%;

[0039] (3) Forge and cog down the purified molybdenum ingots from step (2), and then subject them to circumferential rolling to get molybdenum target ingots, of which the total deformation is 87%. The circumferential rolling is carried out in a manner of continuously changing the rolling direction, and the rolling direction of a consequent pass forms an angle of 150° with that of the previous pass; each 12 passes is one cycle to obtain molybdenum target ingots;

[0040] (4) Subject the molybdenum target ingots from step (3) in a vacuum annealing furnace to anneal at a temperature of 1150℃ and vacuum degree of 1 .5×10-3Pa for 1h; finally, process according to specified specifications to prepare the molybdenum target material.

[0041] Molybdenum target materials obtained by this method has a purity of 99 .999% and density of 99 .9%, with contents of C, N and O less than 5ppm, with uniform particle size of molybdenum target material, mean crystal particle diameter of 60μm and crystallization orientation taking {100}<011> as the preferred texture orientation.

[0042] Test case

[0043] The test case subjects molybdenum target material prepared in embodiment 1 for the preparation method of molybdenum target material to

microscope for observation of microstructure; the result is as shown in Figure 1. From Figure 1 we can see that the molybdenum target material obtained by this method has a purity of 99 .99% and density of 99%, with contents of C, N and O less than 5ppm, with uniform particle size of molybdenum target material, mean crystal particle diameter of 60μm and crystallization orientation taking {100}<011> as the preferred texture orientation.

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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A preparation method of molybdenum target material, characterized in that: it comprises the following steps:

(1) Roll and sinter molybdenum materials to form cylinder molybdenum ingots;

(2) Prepare molybdenum target ingots with cylinder molybdenum ingots from step (1) by circumferential rolling;

(3) Acquire molybdenum target materials with molybdenum target ingots from step (2) by vacuum annealing.

2. Preparation method of molybdenum target material as claim 1, characterized by: the said molybdenum material in step (1) is molybdenum powder with granularity being 2～3μm.

3. Preparation method of molybdenum target material as claim 1 or 2, characterized by: the said molybdenum material in step (1) has a purity ≥99 .95%.

4. Preparation method of molybdenum target material as claim 1, characterized by: the said sintering temperature in step (1) is 1800～1850℃ and sintering duration is 6～8h.

5. Preparation method of molybdenum target material as claim 1, characterized by: the said cylinder molybdenum ingots in step (1) are made into purified cylinder molybdenum ingots by purification with a purity ≥99.99%.

6. Preparation method of molybdenum target material as claim 1, characterized by: for the said circumferential rolling in step (2), the rolling direction of a consequent pass forms an included angle of 120°～150° with that of the previous pass.

7. Preparation method of molybdenum target material as claim 6, characterized by: the said molybdenum target ingots in step (2) have a total deformation of 70～90%.

8. Preparation method of molybdenum target material as claim 1, characterized by: the said vacuum annealing temperature in step (3) is 1050～1150℃ and the duration is 1～2h.

9. Preparation method of molybdenum target material as claim 1, characterized by: the vacuum degree of the said vacuum annealing in step (3) is at least 1 .5×10^{-3} Pa.

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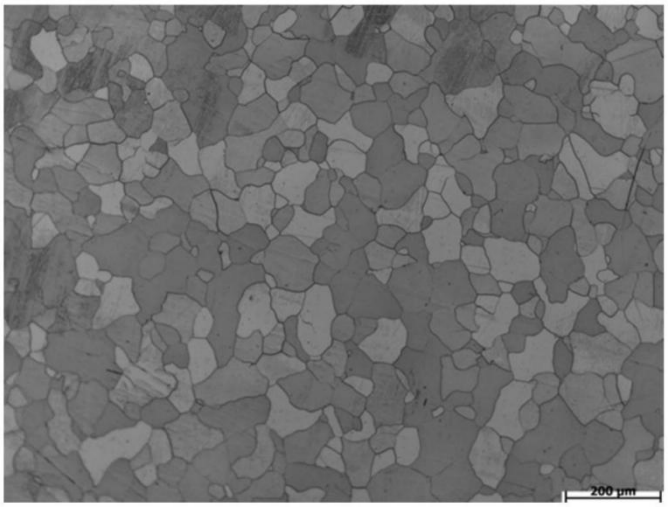

Figure 1