

FERROUS ALLOY SPUTTERING TARGET AND PREPARATION METHOD THEREOF

BACKGROUND OF THE INVENTION

1. Field of the Invention

5 The present invention relates to a ferrous alloy sputtering target and a preparation method thereof, especially to an iron-molybdenum alloy sputtering target and a preparation method thereof.

2. Description of the Prior Arts

 With the rapid development of the technology industry, the capacity demand for
10 digital storage media is getting higher and higher. According to the direction of magnetization of the magnetic head, magnetic recording media can be divided into longitudinal magnetic recording (LMR) media and perpendicular magnetic recording (PMR) media. As the magnetic recording density of longitudinal magnetic recording media has reached physical limitations, the industry mostly aims at material research and
15 development of perpendicular magnetic recording media.

 The perpendicular magnetic recording medium has a layered structure including a substrate, a base layer, a soft underlayer, an intermediate layer, a magnetic recording layer, a lubricant layer and a protection layer from bottom to top. To design materials for perpendicular magnetic recording media, various ferrous alloy sputtering targets have
20 been developed due to the high conductivity of iron. As iron and molybdenum have a good match for their lattice sizes, a ferrous alloy sputtering target mixed with molybdenum component is also developed, aiming at preparing the functional layers of the perpendicular magnetic recording media by sputtering.

 However, molybdenum has a high melting point of 2600°C, resulting in difficulty
25 of sintering for the molybdenum-doped ferrous alloy sputtering targets. To lower the sintering difficulty, doping relatively low melting-point metals (e.g. copper) or reducing the amount of the molybdenum in the ferrous alloy sputtering targets have been tried.

The above approaches somehow ameliorate or alleviate the sintering issues, but lots of undesirable micropores and defects exist in the metallographic structure of the ferrous alloy sputtering targets, resulting in reduced compactness and poor conductivity uniformity of the sputtering targets, and even a higher occurrence of abnormal arcing during the sputtering process, thereby deteriorating sputtering quality. The above approaches also severely restrict the allowable amount of molybdenum in the ferrous alloy sputtering target, and the iron-molybdenum alloy sputtering targets that meet the back-end applications have not yet to be developed. Moreover, the functional layers of iron-molybdenum alloy cannot be readily prepared by directly sputtering the target.

SUMMARY OF THE INVENTION

In view of the above drawbacks, the purpose of the present invention is to reduce the undesirable micropores and defects in the ferrous alloy sputtering targets mixed with molybdenum, and to ameliorate or alleviate the issues of reduced compactness, poor conductivity uniformity, and the frequent occurrence of abnormal arcing during the sputtering process.

To achieve the aforementioned purpose, the present invention provides a ferrous alloy sputtering target, comprising iron (Fe), molybdenum (Mo) and an additive ingredient, wherein the additive ingredient comprises a first additive metal, a second additive metal or a combination thereof, the first additive metal is selected from the group consisting of titanium (Ti), copper (Cu), germanium (Ge) and combinations thereof, the second additive metal is selected from the group consisting of cobalt (Co), manganese (Mn), silver (Ag), gold (Au), palladium (Pd), platinum (Pt), rhodium (Rh) and combinations thereof; based on a total atom number of the ferrous alloy sputtering target, Fe is in an amount of 25 atomic percent (at%) to 55 at%, and the ferrous alloy sputtering target comprises multiple spherical grains, a composition of the spherical grains comprises Fe-Mo alloy, and the spherical grains have an average particle size less than 80 microns (μm).

By means of controlling the overall composition of the ferrous alloy sputtering target, the composition of the spherical grains in the ferrous alloy sputtering target, and the average particle size of Fe-Mo alloy spherical grains, it is conducive to alleviate and inhibit the formation of micropores and defects in the metallographic structure, increase the electrical conductivity uniformity of the ferrous alloy sputtering target, and reduce the occurrences of abnormal arcing during the sputtering process of the ferrous alloy sputtering target.

Further, the present invention also facilitates expanding the flexibility of composition design for the ferrous alloy sputtering targets, and therefore develops both a ferrous alloy sputtering target using iron as the main ingredient (Fe amount > Mo amount) and a ferrous alloy sputtering target using molybdenum as the main ingredient (Mo amount > Fe amount). For these two ferrous alloy sputtering targets, both of them have the advantages of reduced number of micropores and reduced defects in the metallographic structure, good compactness, good conductivity uniformity, and improved sputtering quality.

Optionally, in the ferrous alloy sputtering target, iron may be in an amount greater than, equal to or less than that of molybdenum, and there are no special restrictions. In one embodiment, based on a total atom number of the ferrous alloy sputtering target, iron may be in an amount of 25 at% to 55 at%, and molybdenum may be in an amount of 40 at% to 65 at%. In another embodiment, based on a total atom number of the ferrous alloy sputtering target, iron may be in an amount of 25 at% to 55 at%, and molybdenum may be in an amount of 41 at% to 62 at%. In other embodiments, based on a total atom number of the ferrous alloy sputtering target, iron may be in an amount of 30 at% to 50 at% or 30 at% to 40 at%.

Optionally, the composition of the spherical grains comprises a Fe-Mo-Ti alloy. In one embodiment, based on a total atom number of the ferrous alloy sputtering target, iron may be in an amount of 25 at% to 45 at%, molybdenum may be in an amount of 40

at% to 65 at%, and titanium may be in an amount of 3 at% to 15 at%. Preferably, based on a total atom number of the ferrous alloy sputtering target, iron may be in an amount of 27 at% to 40 at%, molybdenum may be in an amount of 51 at% to 58 at% and titanium may be in an amount of 4 at% to 13 at%. For further clarification, the first additive metal
5 may or may not comprise titanium.

Preferably, based on a total atom number of the ferrous alloy sputtering target, the additive ingredient may be in an amount of 3 at% to 15 at%. In the embodiment that the additive ingredient may be a first additive metal, based on a total atom number of the ferrous alloy sputtering target, the first additive metal may be in an amount of 3 at% to 15
10 at%; in other embodiments, based on a total atom number of the ferrous alloy sputtering target, the first additive metal may be in an amount of 4 at% to 13 at% or 7 at% to 12 at%. In the embodiment that the additive ingredient may be a second additive metal, based on a total atom number of the ferrous alloy sputtering target, the second additive metal may be in an amount of 3 at% to 15 at%; in other embodiments, based on a total
15 atom number of the ferrous alloy sputtering target, the second additive metal may be in an amount of 3 at% to 12 at%, 4 at% to 10 at% or 5 at% to 8 at%. In the embodiment that the additive ingredient may be a combination of a first additive metal and a second additive metal, based on a total atom number of the ferrous alloy sputtering target, the individual amount of the first additive metal may range from 1 at% to 12 at%, the
20 individual amount of the second additive metal may range from 1 at% to 12 at%, and the first additive metal and the second additive metal may be in a total amount of 3 at% to 15 at%.

Further, the additive ingredient may comprise nitride. That is, the ferrous alloy sputtering target may comprise a combination of iron, molybdenum and the first additive
25 metal; a combination of iron, molybdenum and the second additive metal; a combination of iron, molybdenum, the first additive metal and nitride; a combination of molybdenum, iron, the second additive metal and nitride; or a combination of iron, molybdenum, the

first additive metal, the second additive metal and nitride.

In the embodiment that the spherical grains may comprise the Fe-Mo-Ti alloy, the ferrous alloy sputtering target may comprise a combination of iron, molybdenum, titanium and the first additive metal; a combination of iron, molybdenum, titanium and the second additive metal; a combination of iron, molybdenum, titanium, the first additive metal and nitride; a combination of molybdenum, iron, titanium, the second additive metal and nitride; or a combination of iron, molybdenum, titanium, the first additive metal, the second additive metal and nitride.

Preferably, nitride may comprise titanium nitride (TiN), aluminum nitride (AlN), boron nitride (BN), chromium nitride (CrN), zirconium nitride (ZrN), tantalum nitride (TaN), hafnium nitride (HfN), silicon nitride (Si₃N₄), tungsten nitride (WN), vanadium nitride (VN), titanium carbonitride (TiCN) or any combination thereof. Preferably, based on a total atom number of the ferrous alloy sputtering target, the nitride may be in an amount of 1 at% to 10 at%; More preferably, based on a total atom number of the ferrous alloy sputtering target, the nitride may be in an amount of 2 at% to 10 at% or 5 at% to 10 at%.

To achieve the aforementioned purpose, the present invention further provides a preparation method for a ferrous alloy sputtering target, comprising:

subjecting an iron raw material and a molybdenum raw material to atomization granulation to obtain a Fe-Mo alloy atomized powder, and the Fe-Mo alloy atomized powder has a D95 less than 125 μm;

mixing the Fe-Mo alloy atomized powder and an additive raw material to obtain a mixed raw material, and the additive raw material comprises a first additive metal raw material, a second additive metal raw material or a combination thereof, the first additive metal raw material is selected from the group consisting of a titanium raw material, a copper raw material, a germanium raw material and combinations thereof, the second additive metal raw material is selected from the group consisting of a cobalt raw material,

a manganese raw material, a silver raw material, a gold raw material, a palladium raw material, a platinum raw material, a rhodium raw material and combinations thereof, wherein based on a total atom number of the mixed raw material, the iron raw material is in an amount of 25 at% to 55 at%; and

5 sintering the mixed raw material at a temperature from 1000°C to 1400°C to obtain the ferrous alloy sputtering target;

 wherein the ferrous alloy sputtering target comprises multiple spherical grains, a composition of the spherical grains comprises a Fe-Mo alloy, and the spherical grains have an average particle size less than 80 μm .

10 By means of controlling the composition of the mixed raw material, the D95 of the Fe-Mo alloy atomized powder and the sintering temperature, it is conducive to solve the sintering issues resulting from the high melting point of molybdenum to obtain the ferrous alloy sputtering target with refined spherical grains comprising the Fe-Mo alloy. Therefore, it is conducive to alleviate and inhibit the formation of micropores and defects
15 in the metallographic structure, increase the electrical conductivity uniformity of the ferrous alloy sputtering target, and reduce the occurrences of abnormal arcing during the sputtering process of the ferrous alloy sputtering target.

 Optionally, in the mixed raw material, the iron raw material may be in an amount greater than, equal to or less than that of the molybdenum raw material, and there are no
20 special restrictions. In the preparation method for the ferrous alloy sputtering target, in one embodiment, based on a total atom number of the mixed raw material, the iron raw material may be in an amount of 25 at% to 55 at%, and the molybdenum raw material may be in an amount of 40 at% to 65 at%; in another embodiment, based on a total atom number of the mixed raw material, the iron raw material may be in an amount of 25 at%
25 to 55 at%, and the molybdenum raw material may be in an amount of 41 at% to 62 at%. In other embodiments, based on a total atom number of the mixed raw material, the iron raw material may be in an amount of 30 at% to 50 at% or 30 at% to 40 at%.

Optionally, the step of subjecting the iron raw material and the molybdenum raw material to atomization granulation to obtain the Fe-Mo alloy atomized powder comprises: subjecting the iron raw material, the molybdenum raw material, and the titanium raw material to atomization granulation to obtain the Fe-Mo-Ti alloy atomized powder, wherein the Fe-Mo-Ti alloy atomized powder has a D95 less than 125 microns; the step of mixing the Fe-Mo alloy atomized powder and the additive raw material to obtain the mixed raw material comprises: mixing the Fe-Mo-Ti alloy atomized powder and the additive raw material to obtain the mixed raw material; and the composition of the spherical grains of the ferrous alloy sputtering target comprises the Fe-Mo-Ti alloy. That is, the titanium raw material may be further subjected to atomization granulation to obtain a Fe-Mo-Ti alloy atomized powder.

For further clarification, the first additive metal raw material may or may not comprise the titanium raw material. That is, even some of the titanium raw material is subjected to atomization granulation to obtain the Fe-Mo-Ti alloy atomized powder, some of the titanium raw material may still be mixed with the Fe-Mo-Ti alloy atomized powder to obtain the mixed raw material; or all titanium raw material is subjected to atomization granulation to obtain the Fe-Mo-Ti alloy atomized powder, and no titanium raw material is further mixed with the Fe-Mo-Ti alloy atomized powder to obtain the mixed raw material.

In the preparation method for the ferrous alloy sputtering target, in one embodiment, based on a total atom number of the mixed raw material, the iron raw material may be in an amount of 25 at% to 45 at%, the molybdenum raw material may be in an amount of 40 at% to 65 at% , and the titanium raw material may be in an amount of 3 at% to 15 at%; in another embodiment, based on a total atom number of the mixed raw material, the iron raw material may be in an amount of 27 at% to 40 at%, the molybdenum raw material may be in an amount of 51 at% to 58 at%, and the titanium raw material may be in an amount of 4 at% to 13 at%.

Preferably, the Fe-Mo or Fe-Mo-Ti alloy atomized powder may have a D95 greater than or equal to 50 μm and less than 125 μm . Specifically, the Fe-Mo alloy or Fe-Mo-Ti atomized powder may have a D95 greater than or equal to 80 μm and less than 125 μm . Optionally, in a step for the atomization granulation, the molybdenum raw material may have a D95 less than 20 μm , the iron raw material may have a D95 less than 10 μm , and the titanium raw material may have a D95 less than 50 μm , but not limited thereto. In the present invention, D95 refers to the equivalent diameter (particle size) of the largest particle where the cumulative distribution in the particle size distribution (PSD) is 95%, that is, based on the total volume of all particles, the particles with the particle size less than that of D95 may be in an amount of 95 volume %.

Preferably, based on a total atom number of the mixed raw material, the additive raw material may be in an amount of 3 at% to 15 at%. In the embodiment that the additive raw material may be a first additive metal raw material, based on a total atom number of the mixed raw material, the first additive metal raw material may be in an amount of 3 at% to 15 at%; in other embodiments, based on a total atom number of the mixed raw material, the first additive metal raw material may be in an amount of 4 at% to 13 at% or 7 at% to 12 at%. In the embodiment that the additive raw material may be a second additive metal raw material, based on a total atom number of the mixed raw material, the second additive metal raw material may be in an amount of 3 at% to 15 at%; in other embodiments, based on a total atom number of the mixed raw material, the second additive metal raw material may be in an amount of 3 at% to 12 at%, 4 at% to 10 at% or 5 at% to 8 at%. In the embodiment that the additive raw material may be a combination of a first additive metal raw material and a second additive metal raw material, based on a total atom number of the mixed raw material, the individual amount of the first additive metal raw material may range from 1 at% to 12 at%, the individual amount of the second additive metal raw material may range from 1 at% to 12 at%, and the first additive metal raw material and second additive metal raw material may be in an

total amount of 3 at% to 15 at%.

Preferably, the additive raw material may have a D95 less than 50 μm .

Specifically, for serving as the first additive metal raw material, a titanium raw material may have a D95 less than 50 μm , a copper raw material may have a D95 less than 40 μm ,

5 and a germanium raw material may have a D95 less than 50 μm . For serving as the second additive metal raw material, a cobalt raw material may have a D95 less than 30 μm or less than 15 μm , a manganese raw material may have a D95 less than 30 μm or less than 20 μm , a silver raw material may have a D95 less than 15 μm or less than 10 μm , a gold raw material may have a D95 less than 15 μm or less than 10 μm , a palladium
10 raw material may have a D95 less than 75 μm or less than 50 μm , a platinum raw material may have a D95 less than 30 μm or less than 10 μm , a rhodium raw material may have a D95 less than 45 μm or less than 30 μm , but not limited thereto. Optionally, such raw materials of pure metals have a D95 with a lower limit of 0.1 μm , 0.5 μm or 1 μm . That is, a titanium raw material may have a D95 greater than or equal to 0.1 μm and
15 less than 50 μm , a copper raw material may have a D95 greater than or equal to 0.1 μm and less than 40 μm , a germanium raw material may have a D95 greater than or equal to 0.1 μm and less than 50 μm , a cobalt raw material may have a D95 greater than or equal to 0.1 μm and less than 15 μm , a manganese raw material may have a D95 greater than or equal to 0.1 μm and less than 20 μm , a silver raw material may have a D95 greater than
20 or equal to 0.1 μm and less than 10 μm , a gold raw material may have a D95 greater than or equal to 0.1 μm and less than 10 μm , a palladium raw material may have a D95 greater than or equal to 0.1 μm and less than 50 μm , a platinum raw material may have a D95 greater than or equal to 0.1 μm and less than 10 μm , and a rhodium raw material may have a D95 greater than or equal to 0.1 μm and less than 30 μm . According to the present
25 invention, the pure metal raw materials with said specific D95 may be obtained directly by purchase, or by firstly sieving the pure metal raw materials with said specific D95 through a specific mesh, and then be subjected to the subsequent processes to ultimately

further refine the average particle size of the spherical grains which mainly comprises the Fe-Mo alloy or Fe-Mo-Ti alloy in the ferrous alloy sputtering target.

Further, the additive raw material further comprises a nitride raw material. That is, the mixed raw material may comprise a combination of an iron raw material, a molybdenum raw material and a first additive metal raw material, a combination of an iron raw material, a molybdenum raw material and a second additive metal raw material, a combination of an iron raw material, a molybdenum raw material, a first additive metal raw material and a nitride raw material, a combination of an iron raw material, a molybdenum raw material, a second additive metal raw material and a nitride raw material, or a combination of an iron raw material, a molybdenum raw material, a first additive metal raw material, a second additive metal raw material and a nitride raw material.

In the embodiment that the spherical grains may comprise the Fe-Mo-Ti alloy, the mixed raw material may comprise a combination of an iron raw material, a molybdenum raw material, a titanium raw material and a first additive metal raw material, a combination of an iron raw material, a molybdenum raw material, a titanium raw material and a second additive metal raw material, a combination of an iron raw material, a molybdenum raw material, a titanium raw material, a first additive metal raw material and a nitride raw material, a combination of an iron raw material, a molybdenum raw material, a titanium raw material, a second additive metal raw material and a nitride raw material, or a combination of an iron raw material, a molybdenum raw material, a titanium raw material, a first additive metal raw material, a second additive metal raw material and a nitride raw material.

Preferably, the nitride raw material may comprise a titanium nitride raw material, an aluminum nitride raw material, a boron nitride raw material, a chromium nitride raw material, a zirconium nitride raw material, a tantalum nitride raw material, a hafnium nitride raw material, a silicon nitride raw material, a tungsten nitride raw material, a

vanadium nitride raw material, a titanium carbonitride raw material or a combination thereof. Preferably, based on a total atom number of the mixed raw material, the nitride raw material may be in an amount of 1 at% to 10 at%; more preferably, based on a total atom number of the mixed raw material, the nitride raw material may be in an amount of 2 at% to 10 at% or 5 at% to 10 at%.

Preferably, the nitride raw material may have a D95 less than 5 μm . Specifically, a titanium nitride raw material may have a D95 less than 5 μm , an aluminum nitride raw material may have a D95 less than 3 μm , a boron nitride raw material may have a D95 less than 3 μm , a chromium nitride raw material may have a D95 less than 5 μm , a zirconium nitride raw material may have a D95 less than 3 μm , a tantalum nitride raw material may have a D95 less than 5 μm , a hafnium nitride raw material may have a D95 less than 5 μm , a silicon nitride raw material may have a D95 less than 3 μm , a tungsten nitride raw material may have a D95 less than 5 μm , a vanadium nitride raw material may have a D95 less than 5 μm , and a titanium carbonitride raw material may have a D95 less than 3 μm . Optionally, the nitride raw material may have a D95 with a lower limit of 0.1 μm , 0.5 μm or 1 μm . That is, a titanium nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 5 μm , an aluminum nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 3 μm , a boron nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 3 μm , a chromium nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 5 μm , a zirconium nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 3 μm , a tantalum nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 5 μm , a hafnium nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 5 μm , a silicon nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 3 μm , a tungsten nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 5 μm , a vanadium nitride raw material may have a D95 greater than or equal to 0.1 μm and less than 5 μm , and a titanium

carbonitride raw material may have a D95 greater than or equal to 0.1 μm and less than 3 μm . According to the present invention, the nitride raw materials with said specific D95 may be obtained directly by purchase, or by firstly sieving the nitride raw materials comprising said specific D95 through a specific mesh, and then be subjected to the subsequent processes to ultimately further refine the average particle size of the spherical grains which mainly comprises the Fe-Mo alloy or Fe-Mo-Ti alloy in the ferrous alloy sputtering target.

Preferably, the preparation method for the ferrous alloy sputtering target of the present invention comprises sintering the mixed raw material under a temperature from 1000°C to 1400°C and a pressure of 1200 kgf/cm² to 1800 kgf/cm² to obtain the ferrous alloy sputtering target.

Preferably, in the ferrous alloy sputtering target, the spherical grains may have an average particle size greater than or equal to 20 μm and less than 80 μm . More preferably, the spherical grains may have an average particle size of 30 μm to 75 μm . In one embodiment, in the ferrous alloy sputtering target, the spherical grains may have a composition consisting of the Fe-Mo alloy or Fe-Mo-Ti alloy.

Preferably, the ferrous alloy sputtering target may have oxygen in an amount less than 900 parts per million (ppm). Specifically, the ferrous alloy sputtering target may have oxygen in an amount greater than or equal to 300 ppm and less than 900 ppm. In one embodiment, the ferrous alloy sputtering target may have oxygen in an amount greater than or equal to 500 ppm and less than 900 ppm.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is the photo at 500x magnification of the metallographic structure of the ferrous alloy sputtering target of Comparative example 2 and is further marked to demonstrate the phase composition therein.

FIG. 2A is the photo at 200x magnification of the metallographic structure of the ferrous alloy sputtering target of Example 3, and is further marked to provide four

analysis lines for analyzing the average particle size of spherical grains and the numbers of 14 spherical grains therein.

FIG. 2B is the photo at 200x magnification of the metallographic structure of the ferrous alloy sputtering target of Comparative example 9, and is further marked to provide four analysis lines for analyzing the average particle size of spherical grains and the numbers of 8 spherical grains therein.

FIG. 3A is the photo at 2000x magnification of the metallographic structure of the ferrous alloy sputtering target of Example 3, and is further marked to circle the sites of 10 micropores therein.

FIG. 3B is the photo at 2000x magnification of the metallographic structure of the ferrous alloy sputtering target of Example 11, and is further marked to circle the sites of 3 micropores therein.

FIG. 3C is the photo at 2000x magnification of the metallographic structure of the ferrous alloy sputtering target of Comparative example 2, and is further marked to circle the sites of 59 micropores therein.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Several ferrous alloy sputtering targets and the preparation methods thereof are exemplified below, and the embodiments of the present invention will be described in detail. People skilled in the art may easily understand the advantages and effects of the present invention through the disclosure of the present specification, and conduct various modifications and adjustments without deviating from the spirit of the present invention to implement or carry out the present invention.

Examples 1 to 21 (E1 to E21)

All raw material powders were provided according to the composition of the ferrous alloy sputtering target shown in Table 1. In Table 1, the composition of the ferrous alloy sputtering target may be demonstrated by the formula of $a\text{Fe}-b\text{Mo}-c1\text{Ti}-c2\text{Cu}-c3\text{Ge}-$

d1Co-d2Mn-d3Ag-d4Au-d5Pd-d6Pt-d7Rh-e1TiN-e2AlN-e3BN-e4CrN-e5ZrN-e6TaNe7HfN-e8Si₃N₄-e9WN-e10VN-e11TiCN; wherein “a” indicates the amount of Fe based on the total atom number of the ferrous alloy sputtering target; “b” indicates the amount of Mo based on the total atom number of the ferrous alloy sputtering target; “c1”, “c2”, and “c3” respectively indicate the amount of titanium (Ti), copper (Cu), germanium (Ge) based on the total atom number of the ferrous alloy sputtering target; “d1”, “d2”, “d3”, “d4”, “d5”, “d6”, and “d7” respectively indicate the amount of cobalt (Co), manganese (Mn), silver (Ag), gold (Au), palladium (Pd), platinum (Pt), rhodium (Rh) based on the total atom number of the ferrous alloy sputtering target; “e1”, “e2”, “e3”, “e4”, “e5”, “e6”, “e7”, “e8”, “e9”, “e10”, and “e11” respectively indicate the amount of titanium nitride (TiN), aluminum nitride (AlN), boron nitride (BN), chromium nitride (CrN), zirconium nitride (ZrN), tantalum nitride (TaN), hafnium nitride (HfN), silicon nitride (Si₃N₄), tungsten nitride (WN), vanadium nitride (VN), titanium carbonitride (TiCN) based on the total atom number of the ferrous alloy sputtering target, and the units thereof are at%.

The iron raw material powder had a D95 less than 10 μm, the molybdenum raw material powder and the manganese raw material powder had a D95 less than 20 μm, the titanium raw material powder and the germanium raw material powder had a D95 less than 50 μm, the copper raw material powder had a D95 less than 40 μm, the cobalt raw material powder had a D95 less than 15 μm, the silver raw material powder and vanadium nitride raw material had a D95 less than 10 μm, the titanium nitride raw material powder had a D95 less than 5 μm, and the aluminum nitride raw material powder had a D95 less than 3 μm. The pure metal raw materials or the nitride raw material powder with said specific D95 were sieved through a mesh (#300) to remove undesirable large particles, and then subjected to the subsequent processes.

After the required raw material powders were ready, according to the compositions of Example 1 to Example 18 shown in Table 1, both the molybdenum raw material

powder with a D95 less than 20 μm and the iron raw material powder with a D95 less than 10 μm in appropriate amounts were subjected to spray granulation to form a Fe-Mo alloy atomized powder. For Example 19 to Example 21, the molybdenum raw material powder, the iron raw material powder and the titanium raw material powder in

5 appropriate amounts were subjected to spray granulation to form a Fe-Mo-Ti alloy atomized powder. The D95 of the Fe-Mo or Fe-Mo-Ti alloy atomized powders (abbreviated as D95 of Alloy) in Example 1 to Example 21 was less than 125 μm , and the respective specific values were shown in Table 1.

Next, according to the compositions of Example 1 to Example 21 shown in Table 1,
10 the Fe-Mo or Fe-Mo-Ti alloy atomized powders with specific particle sizes along with the pure metal raw material powders and/or the nitride raw material powders in an appropriate amount were sieved through a mesh (#60) and mixed to obtain raw material powders of Example 1 to Example 21. The raw material powders and grinding balls in a weight ratio of 1:1 were put into a ball mill tank, and milled at a rotation speed of 180
15 rpm for 1 hour in a ball mill (model: PMU-4-815, purchased from ACPA Technology Co. Ltd.) for uniform mixing to obtain the mixed raw material powders of Example 1 to Example 21.

Next, the mixed raw material powders were filled in a can, and pre-pressed at a pressure of 1000 psi by a hydraulic press for shaping. The compact was then subjected to
20 hot isostatic pressing (HIP) for consecutive sintering under a temperature of about 1000°C to 1400°C and a pressure of about 1200 kgf/cm² to 1800 kgf/cm² for 1 hour to 3 hours. After sintering, the sintered compact was further processed by wire cutting, turning on a lathe and grinding to obtain a circular sputtering target with an outer diameter of 180 mm and a thickness of 8.5 mm. Then the ferrous alloy sputtering targets of Example 1 to
25 Example 21 were obtained. The parameters of the sintering temperature and sintering pressure in preparing the ferrous alloy sputtering targets of Example 1 to Example 21 were shown in Table 1.

Comparative examples 1 to 3 (C1 to C3)

The preparation methods of Comparative examples 1 to 3 were different from those of Examples 1 to 21, wherein the Fe-Mo alloy atomized powders with specific particle sizes of Comparative examples 1 to 3 were obtained by directly mixing the pure metal raw material powders without spray granulation. The specific preparation process conditions were described below.

The compositions of the ferrous alloy sputtering targets of Comparative examples 1 to 3 were demonstrated in Table 1 by the formula of $a\text{Fe}-b\text{Mo}-c\text{Ti}$. Before mixing the raw material powders, the iron raw material powder with a D95 less than 10 μm , the molybdenum raw material powder with a D95 less than 20 μm , and the titanium raw material powder with a D95 less than 50 μm were sieved through a mesh (#300), further sieved through a mesh (#60) and mixed in appropriate amounts according to the compositions shown in Table 1 to obtain raw material powders of Comparative examples 1 to 3. The raw material powders and grinding balls in a weight ratio of 1 :1 were put into a ball mill tank, and subjected to ball milling at a rotation speed of 180 rpm for 1 hour in a ball mill (model: PMU-4-815, purchased from ACPA Technology Co. Ltd.) for even mixing to obtain the mixed raw material powders of Comparative examples 1 to 3.

Next, the mixed raw material powders were filled in a can, pre-pressed at a pressure of 1000 psi by a hydraulic press for shaping. The compact was then subjected to HIP for consecutive sintering under a temperature of about 1000°C to 1400°C and a pressure of about 1200 kgf/cm² to 1800 kgf/cm² for 1 to 3 hours. After sintering, the sintered compact was further processed by wire cutting, turning on a lathe and grinding to obtain a circular sputtering target with an outer diameter of 180 mm and a thickness of 8.5 mm. Then, the ferrous alloy sputtering targets of Comparative examples 1 to 3 were obtained. The parameters of the sintering temperature and sintering pressure in preparing the ferrous alloy sputtering targets of Comparative examples 1 to 3 were shown in Table 1.

For clarification, the Fe-Mo alloy atomized powders with specific particle sizes of

Comparative examples 1 to 3 were obtained without spray granulation, so the “D95 of Alloy” in Table 1 were recorded as “N.A.” (not available).

Comparative examples 4 to 12 (C4 to C12)

The preparation methods of Comparative examples 4 to 12 were different from those of Examples 1 to 21, wherein the preparation methods for Comparative examples 4 to 12 also carried out the spray granulation steps to obtain the Fe-Mo alloy atomized powders, but the Fe-Mo alloy atomized powders of Comparative examples 4 to 12 had larger particle sizes than those of Examples 1 to 21.

First, the compositions of the ferrous alloy sputtering targets of Comparative examples 4 to 12 were shown in Table 1 and demonstrated by the formula of $a\text{Fe}-b\text{Mo}-c1\text{Ti}-c2\text{Cu}-d1\text{Co}-d2\text{Mn}-e2\text{AlN}$. The iron raw material powder had a D95 less than 10 μm , the molybdenum raw material powder had a D95 less than 20 μm , the titanium raw material powder had a D95 less than 50 μm , the copper raw material powder had a D95 less than 40 μm , the cobalt raw material powder had a D95 less than 15 μm , the manganese raw material powder had a D95 less than 20 μm , and the aluminum nitride raw material powder had a D95 less than 3 μm . The pure metal raw materials or the nitride raw material powder were respectively sieved through a mesh (#300). Further, according to the compositions shown in Table 1, both the molybdenum raw material powder and the iron raw material powder in appropriate amounts were subjected to spray granulation to form a Fe-Mo alloy atomized powder; wherein the D95 of the Fe-Mo alloy atomized powders (abbreviated as D95 of Alloy) in Comparative examples 4 to 12 were about 140 μm to 250 μm , and the respective specific values were shown in Table 1.

Next, according to the compositions shown in Table 1, the Fe-Mo alloy atomized powders with specific particle sizes along with the pure metal raw material powders of titanium, copper, cobalt and manganese in an appropriate amount were sieved through a mesh (#60) and mixed to obtain raw material powders of Comparative examples 4 to 12. The raw material powders and grinding balls in a weight ratio of 1 :1 were put into a ball

mill tank, and subjected to ball milling at a rotation speed of 180 rpm for 1 hour in a ball mill (model: PMU-4-815, purchased from ACPA Technology Co. Ltd.) for even mixing to obtain the mixed raw material powders of Comparative examples 4 to 12.

Next, the mixed raw material powders were filled in a can, pre-pressed at a pressure of 1000 psi by a hydraulic press for shaping. The compact was then subjected to HIP for consecutive sintering under a temperature of about 1000°C to 1400°C and a pressure of about 1200 kgf/cm² to 1800 kgf/cm² for 1 to 3 hours. After sintering, the sintered compact was further processed by wire cutting, turning on a lathe and grinding to obtain a circular sputtering target with an outer diameter of 180 mm and a thickness of 8.5 mm.

Then, the ferrous alloy sputtering targets of Comparative examples 4 to 12 were obtained. The parameters of the sintering temperature and sintering pressure in preparing the ferrous alloy sputtering targets of Comparative examples 4 to 12 were shown in Table 1.

Comparative examples 13 to 18 (C13 to C18)

The preparation methods of Comparative examples 13 to 18 were different from those of Examples 1 to 18, wherein the preparation methods for Comparative examples 13 to 18 also carried out the spray granulation steps to obtain the Fe-Mo alloy atomized powders, and the Fe-Mo alloy atomized powders of Comparative examples 13 to 18 had significantly larger particle sizes than those of Examples 1 to 18. Further, the pure metal raw materials and the nitride raw material powder of Comparative examples 13 to 18 were not sieved to remove undesirable large particles. The specific preparation process conditions were described below.

First, the compositions of the ferrous alloy sputtering targets of Comparative examples 13 to 18 were demonstrated in Table 1 by the formula of aFe-bMo-c1Ti-d3Ag-e1TiN. The iron raw material powder had a D95 less than 10 μm, the molybdenum raw material powder and the manganese raw material powder had a D95 less than 20 μm, the titanium raw material powder and the germanium raw material powder had a D95 less than 50 μm, the silver raw material powder had a D95 less than 10 μm, and the titanium

nitride raw material powder had a D95 less than 5 μm . The pure metal raw materials or the nitride raw material powder were not sieved through a mesh (#300). According to the compositions shown in Table 1, both the molybdenum raw material powder and the iron raw material powder in appropriate amounts were subjected to spray granulation to form a Fe-Mo alloy atomized powder; wherein the D95 of the Fe-Mo alloy atomized powders (abbreviated as D95 of Alloy) in Comparative examples 13 to 18 were greater than 250 μm , and the respective specific values were shown in Table 1.

Next, according to the compositions shown in Table 1, the Fe-Mo alloy atomized powders with specific particle sizes along with the pure metal raw material powders of titanium and silver and a titanium nitride raw material powder in an appropriate amount were sieved through a mesh (#60) and mixed to obtain raw material powders of Comparative examples 13 to 18. The raw material powders and grinding balls in a weight ratio of 1 :1 were put into a ball mill tank, and subjected to ball milling at a rotation speed of 180 rpm for 1 hour in a ball mill (model: PMU-4-815, purchased from ACPA Technology Co. Ltd.) for even mixing to obtain the mixed raw material powders of Comparative examples 13 to 18.

Next, the mixed raw material powders were filled in a can, pre-pressed at a pressure of 1000 psi by a hydraulic press for shaping. The compact was then subjected to HIP for consecutive sintering under a temperature of about 1000°C to 1400°C and a pressure of about 1200 kgf/cm² to 1800 kgf/cm² for 1 to 3 hours. After sintering, the sintered compact was further processed by wire cutting, turning on a lathe and grinding to obtain a circular sputtering target with an outer diameter of 180 mm and a thickness of 8.5 mm. Then, the ferrous alloy sputtering targets of Comparative examples 13 to 18 were obtained. The parameters of the sintering temperature and sintering pressure in preparing the ferrous alloy sputtering targets of Comparative examples 13 to 18 were shown in Table 1.

Test example 1: the oxygen amount in the targets

The circular ferrous alloy sputtering targets of Examples 1 to 21 and Comparative examples 1 to 18 were wire cut and ground to obtain pieces with a size of about 5 mm × 20 mm × 2 mm as test samples, and further analyzed by Oxygen/Nitrogen Analyzer (model: EMGA-620W, purchased from Horiba Co., Ltd.) In this test example, the test sample of the specific size were placed in a graphite crucible, heated and burned in an atmosphere with continuous helium flow, so that the oxygen in the test samples would be converted into carbon monoxide and detected by the non-dispersive infrared absorption method to obtain the oxygen amounts of each test sample. The results were shown in Table 1.

In the comparison of the oxygen amounts in the ferrous alloy sputtering targets of Examples 1 to 21 and Comparative Examples 1 to 18, the ferrous alloy sputtering targets of Examples 1 to 21 had oxygen in an amount less than 900 ppm. The ferrous alloy sputtering targets of Comparative examples 1 to 18 had oxygen in an amount greater than 900 ppm. Particularly, the ferrous alloy sputtering target of Comparative example 16 had oxygen in an amount as high as 1847 ppm. Therefore, by controlling the composition of the ferrous alloy sputtering target, and using the Fe-Mo alloy atomized powder with specific D95 for the following mixing and sintering steps in the preparation process, it is conducive to reduce the oxygen amount in the ferrous alloy sputtering targets.

Test example 2: the metallographic structure analysis

The circular ferrous alloy sputtering targets of Examples 1 to 21 and Comparative examples 1 to 18 were wire cut at a distance of about half radius ($\frac{1}{2}r$) from the center to the circumference, ground and polished to obtain pieces in a size of about 10mm×10mm×5mm as test samples, and further observed with a scanning electron microscope (model: SE-3400N, purchased from Hitachi) at 200x, 500x, and 2000x magnification, and photographed to obtain the photos of metallographic structure of the ferrous alloy sputtering targets of Examples 1 to 21 and Comparative examples 1 to 18 for subsequent analysis of the composition and metallographic structure.

At 500x magnification, the spherical grains observed in the metallographic structure photos were selected and detected by energy dispersive X-ray Spectrometer (EDX) for composition analysis to identify the phase composition of the spherical grains in the ferrous alloy sputtering targets. As shown in Table 1, the spherical grains of the ferrous alloy sputtering targets of Examples 1 to 18 and Comparative example 4 to 18 all had a phase composition of Fe-Mo alloy, and the spherical grains of the ferrous alloy sputtering targets of Examples 19 to 21 all had a phase composition of Fe-Mo-Ti alloy and Fe-Mo alloy. Further, there was no spherical grain identified in the ferrous alloy sputtering targets of Comparative examples 1 to 3, so the phase compositions of the spherical grains thereof were recorded as “N.A.” (not available) in Table 1. For convenience of illustration, Comparative example 2 was exemplified here.

As shown in FIG. 1, there was no spherical grain identified in the ferrous alloy sputtering target of Comparative example 2. According to the EDX analysis for the phase compositions at different sites, the ferrous alloy sputtering target of Comparative example 2 had pure iron phase, pure molybdenum phase, Fe-Mo alloy phase and pure titanium phase. Therefore, whether the Fe-Mo alloy atomized powder was firstly prepared for the following mixing and sintering steps in the preparation process demonstrated a substantial impact on the formation of spherical grains and its phase composition in the ferrous alloy sputtering targets.

Further, the intercept method (also called line intercept method) was used to draw four analysis lines (two diagonal lines, a horizontal line and a vertical line) on the metallographic structure photo at 200x magnification, and the four analysis lines all passed through the center of the metallic structure photo. The Fe-Mo alloy spherical grains that fell on the four analysis lines were numbered, and the size (Feret’s Diameter) thereof was measured by the built-in “Analyze” function in Image J to obtain the average particle size of the Fe-Mo alloy spherical grains. The results were shown as “the average particle size of spherical grains” in Table 1. The ferrous alloy sputtering targets of

Example 3 and Comparative example 9 were exemplified here. The metallographic structure photos thereof at 200x magnification marked with four analysis lines along with the numbered spherical grains were shown in FIG. 2A and FIG. 2B. In FIG. 2A, the 14 spherical grains on the four analysis lines were measured by the built-in “Analyze” function in Image J to obtain the sizes of Fe-Mo alloy spherical grains, and the average particle size of Fe-Mo alloy spherical grains of the ferrous alloy sputtering target of Example 3 was 56.92 μm . In FIG. 2B, the eight spherical grains on the four analysis lines were analyzed by the same method, and the average particle size of Fe-Mo alloy spherical grains of the ferrous alloy sputtering target of Comparative example 9 was 88.77 μm . For clarification, there were no spherical grains identified in the ferrous alloy sputtering targets of Comparative examples 1 to 3, so the average particle sizes of Fe-Mo alloy spherical grains in the ferrous alloy sputtering targets thereof were recorded as 0 in Table 1.

In the comparison of the average particle sizes of Fe-Mo alloy spherical grains in the ferrous alloy sputtering targets of Examples 1 to 21 and Comparative Examples 1 to 18, the Fe-Mo alloy spherical grains in the ferrous alloy sputtering targets of Examples 1 to 18 had an average particle size less than 80 μm . There was no spherical grain in the ferrous alloy sputtering targets of Comparative examples 1 to 3. The Fe-Mo alloy spherical grains in the ferrous alloy sputtering targets of Comparative examples 6 to 18 had an average particle size greater than 80 μm . Therefore, the Fe-Mo alloy atomized powder with a specific D95 firstly prepared for the following mixing and sintering steps in the preparation process demonstrated a substantial impact on the average particle size of the Fe-Mo alloy spherical grains in the ferrous alloy sputtering targets.

Further, the micropores in the microstructure of the metallographic structure on the metallographic structure photo at 200x magnification were observed and counted by visual inspection, and the results were recorded in “micropores in the metallographic structure” in Table 1. The ferrous alloy sputtering targets of Example 3, Example 11 and

Comparative example 2 were exemplified here. The micropores on the metallographic structure photo at 2000x magnification were circled, and the results were shown in FIG. 3A to FIG. 3C.

As shown in FIG. 3A and FIG. 3B, the ferrous alloy sputtering targets of Examples 3 and 11 respectively had 10 and 3 micropores in the metallographic structures thereof. As shown in FIG. 3C, the ferrous alloy sputtering target of Comparative example 2 had 59 micropores in the metallographic structures thereof.

According to the microstructure analysis results of the metallographic structure, controlling the D95 of the Fe-Mo alloy atomized powder firstly for the following mixing and sintering steps in the preparation process demonstrated a substantial impact on the average particle size of the spherical grains in the ferrous alloy sputtering target and the numbers of micropores in the metallographic structure.

Further, molybdenum has a high melting point and is not easy to sinter, without the use of Fe-Mo alloy atomized powder of the specific size for preparing the ferrous alloy sputtering target, the sintering issues of insufficient compactness cannot be alleviated, which results in more micropores formed in the pure molybdenum phase, and deteriorates the quality of the ferrous alloy sputtering targets.

In the comparison of micropore defects in the metallographic structures of the ferrous alloy sputtering targets of Examples 1 to 21 and Comparative Examples 1 to 18, the micropore numbers in the metallographic structures of the ferrous alloy sputtering targets of Examples 1 to 21 were less than 20. The micropore numbers in the metallographic structures of the ferrous alloy sputtering targets of Comparative examples 1 to 18 were 35 or more. Particularly, the ferrous alloy sputtering target of Comparative example 2 had as high as 59 micropores in the metallographic structure. Therefore, by any of controlling the composition of the ferrous alloy sputtering target along with the phase composition and average particle size of the spherical grains, or using the Fe-Mo alloy atomized powder with a specific D95 with the addition of specific additive raw

materials, and sintering at a specific temperature, it is conducive to alleviate and inhibit the formation of micropores and defects in the metallographic structure, and increase the compactness of the ferrous alloy sputtering targets.

Test example 3: Conductivity uniformity analysis

5 The conductivity values (unit: %IACS) at 5 points, which were the center of the circular ferrous alloy sputtering targets of Examples 1 to 21 and Comparative examples 1 to 18, and four points about $\frac{2}{3}$ radius ($\frac{2}{3}r$) upward, downward, left, and right from the center, were measured by a conductivity meter (Model: SigmaCheck, purchased from Ether NDE), and the formula of the statistical standard deviation (σ) was used to obtain
10 the conductivity uniformity of the ferrous alloy sputtering target at said 5 different points. The results were shown in Table 1.

As shown in Table 1, the conductivity uniformity of the ferrous alloy sputtering targets of Examples 1 to 21 were less than 5.00×10^{-1} . The conductivity uniformity of the ferrous alloy sputtering targets of Comparative examples 1 to 18 was significantly greater
15 than 5.00×10^{-1} . Particularly, the conductivity uniformity of the ferrous alloy sputtering target of Comparative example 3 was as high as 9.74×10^{-1} . Therefore, by any of controlling the composition of the ferrous alloy sputtering target along with the phase composition and average particle size of the spherical grains, or using the Fe-Mo alloy atomized powder with a specific D95 with the addition of specific additive raw materials,
20 and sintering at a specific temperature, it is conducive to increase the conductivity uniformity of the ferrous alloy sputtering target.

Test example 4: Abnormal arcing test

The circular ferrous alloy sputtering targets of Examples 1 to 21 and Comparative examples 1 to 18 were sputtered by a magnetron sputtering machine in an argon
25 atmosphere with a flow rate of 50 standard cubic centimeters per minute (sccm) and a pressure of 1.05×10^{-2} Tor. The pre-sputtering at 200 watts was carried out for 5 minutes to remove surface dirt, and the test sputtering at 500 watts was carried out for 30 minutes,

wherein the number of occurrences of abnormal arcing in each sample during the test sputtering was calculated. The results were shown in Table 1.

As shown in Table 1, the numbers of occurrences of abnormal arcing in the ferrous alloy sputtering targets of Examples 1 to 21 were less than 50. The numbers of occurrences of abnormal arcing in the ferrous alloy sputtering targets of Comparative examples 1 to 18 were significantly higher than 50. Particularly, the number of occurrences of abnormal arcing in the ferrous alloy sputtering target of Comparative example 16 was as high as 109. Therefore, by controlling the composition of the ferrous alloy sputtering target along with the phase composition and average particle size of the spherical grains, or using the Fe-Mo alloy atomized powder with a specific D95 with the addition of specific additive raw materials, and sintering at a specific temperature, it is conducive to reduce the occurrences of abnormal arcing during the sputtering process of the ferrous alloy sputtering target.

Table 1: the composition of the ferrous alloy sputtering targets of Examples 1 to 21 (E1 to E21) and Comparative Examples 1 to 18 (C1 to C18), the D95 of the Fe-Mo alloy atomized powders (abbreviated as D95 of Alloy) used in the preparation, sintering temperature and pressure (P), oxygen amount, the composition and average particle size of the spherical grains, the number of micropores, conductivity uniformity, and the abnormal arcing times

	Composition of ferrous alloy sputtering target	D95 of Alloy (μm)	Sintering		Oxygen amount (ppm)	Spherical grains		Number of micropores	Conductivity uniformity	Abnormal arcing times
			Temp ($^{\circ}\text{C}$)	P (kgf/cm^2)		Composition	Size (μm)			
E1	30Fe-62Mo-8Ti	108.16	1300	1650	782	Fe-Mo	34.41	18	7.85×10^{-2}	44
E2	34Fe-59Mo-7Ti	110.81	1250	1600	689	Fe-Mo	46.16	11	2.92×10^{-1}	34
E3	33Fe-58Mo-9Ti	119.38	1200	1450	712	Fe-Mo	56.92	10	4.15×10^{-1}	36
E4	38Fe-52Mo-10Ti	109.84	1150	1300	697	Fe-Mo	41.51	9	1.14×10^{-1}	32
E5	35Fe-53Mo-12Ti	110.35	1150	1200	648	Fe-Mo	46.84	5	3.32×10^{-1}	30
E6	45Fe-50Mo-5Ti	109.16	1150	1300	564	Fe-Mo	43.17	6	2.34×10^{-1}	24
E7	33Fe-56Mo-11Ti	121.35	1200	1350	752	Fe-Mo	59.96	7	4.18×10^{-1}	41
E8	28Fe-57Mo-15Ti	112.88	1200	1300	816	Fe-Mo	50.18	8	3.45×10^{-1}	46
E9	33Fe-60Mo-7Cu	110.49	1250	1400	714	Fe-Mo	45.94	14	2.91×10^{-1}	38
E10	35Fe-59Mo-6Ge	115.75	1300	1600	657	Fe-Mo	53.34	13	3.81×10^{-1}	33

E11	55Fe-41Mo-4Ti	111.81	1150	1400	541	Fe-Mo	49.19	3	3.45×10^{-1}	22
E12	25Fe-62Mo-13Ti	113.7	1300	1500	884	Fe-Mo	52.15	17	3.76×10^{-1}	48
E13	38Fe-59Mo-3Ti	124.11	1350	1700	875	Fe-Mo	78.53	15	4.97×10^{-1}	43
E14	37Fe-57Mo-5Ti-1TiN	112.27	1300	1650	862	Fe-Mo	47.35	6	2.25×10^{-1}	28
E15	34Fe-53Mo-3Ti-10AlN	116.13	1300	1800	829	Fe-Mo	53.48	9	1.82×10^{-1}	29
E16	32Fe-62Mo-6Co	118.41	1250	1600	765	Fe-Mo	57.83	12	3.98×10^{-1}	38
E17	34Fe-58Mo-8Mn	122.48	1250	1550	791	Fe-Mo	65.34	15	4.19×10^{-1}	40
E18	36Fe-59Mo-5Ag	114.19	1250	1450	814	Fe-Mo	52.48	13	3.75×10^{-1}	42
E19	38Fe-56Mo-6Ti	101.18	1100	1400	706	Fe-Mo-Ti Fe-Mo	33.51	4	1.05×10^{-1}	18
E20	30Fe-55Mo-9Ti-6Mn	102.67	1200	1500	829	Fe-Mo-Ti Fe-Mo	39.17	5	1.09×10^{-1}	19
E21	34Fe-53Mo-11Ti-2VN	111.15	1300	1600	871	Fe-Mo-Ti Fe-Mo	37.56	6	1.27×10^{-1}	20
C1	23Fe-61Mo-16Ti	NA	1300	1500	1309	N.A.	0	58	9.45×10^{-1}	98
C2	33Fe-58Mo-9Ti	NA	1350	1700	1284	N.A.	0	59	8.96×10^{-1}	92

C3	48Fe-43Mo-9Ti	NA	1150	1300	1279	N.A.	0	51	9.74×10^{-1}	88
C4	23Fe-65Mo-12Ti	157.28	1250	1650	1193	Fe-Mo	78.09	52	6.41×10^{-1}	86
C5	56Fe-41Mo-3Ti	165.91	1200	1400	954	Fe-Mo	79.16	35	8.06×10^{-1}	65
C6	44Fe-54Mo-2Ti	204.62	1250	1600	1159	Fe-Mo	83.84	40	7.64×10^{-1}	84
C7	32Fe-52Mo-16Co	216.52	1250	1600	1214	Fe-Mo	86.61	45	8.08×10^{-1}	84
C8	25Fe-58Mo-17Mn	209.25	1300	1600	1158	Fe-Mo	84.13	41	7.67×10^{-1}	78
C9	40Fe-54Mo-6Ti	221.95	1250	1500	975	Fe-Mo	88.77	38	7.36×10^{-1}	68
C10	31Fe-58Mo-11Cu	194.51	1300	1650	1085	Fe-Mo	81.80	41	6.13×10^{-1}	79
C11	28Fe-55Mo-6Ti-11AlN	209.75	1350	1700	1691	Fe-Mo	83.95	41	8.14×10^{-1}	97
C12	31Fe-58Mo-11Ti	212.64	1350	1650	981	Fe-Mo	84.80	42	7.39×10^{-1}	71
C13	22Fe-68Mo-10Ti	254.82	1400	1800	1248	Fe-Mo	97.93	56	8.81×10^{-1}	86
C14	24Fe-64Mo-12Ti	255.81	1350	1650	1274	Fe-Mo	90.32	50	8.85×10^{-1}	85
C15	35Fe-63Mo-2Ti	263.81	1400	1750	1213	Fe-Mo	102.05	47	9.08×10^{-1}	80
C16	27Fe-59Mo-2Ti-12TiN	251.25	1400	1800	1847	Fe-Mo	88.74	46	9.49×10^{-1}	109
C17	41Fe-57Mo-2Ag	259.17	1300	1550	1191	Fe-Mo	91.66	47	8.49×10^{-1}	81
C18	34Fe-53Mo-13Ti	253.55	1300	1550	1156	Fe-Mo	92.62	36	8.52×10^{-1}	81

Results and discussion

According to the results of the test examples 1 to 4, by controlling the composition and sources of the mixed raw materials (using particular additive raw materials and Fe-Mo alloy atomized powder), the D95 of the Fe-Mo alloy atomized powder, the appropriate sintering temperatures, it is conducive to the preparation of the ferrous alloy sputtering target having specific composition and fined spherical Fe-Mo alloy grains. Therefore, the present invention can alleviate and inhibit the formation of micropores and defects in the metallographic structure, increase the conductivity uniformity of the sputtering target, and lower the number of occurrences of abnormal arcing during sputtering, thereby comprehensively improving the overall quality of the ferrous alloy sputtering target. Further, the ferrous alloy sputtering targets of Examples 1 to 21 had the advantage of low oxygen amount, which is further conducive to improve the quality of the ferrous alloy sputtering targets.

In contrast, the preparation methods of Comparative examples 1 to 3 did not use the Fe-Mo alloy atomized powder and failed to control the D95 of the Fe-Mo alloy atomized powder, so that the composition of the spherical grain thereof is not Fe-Mo alloy. Further, in comparison with the ferrous alloy sputtering targets of Examples 1 to 18, those of Comparative examples 1 to 3 significantly had more oxygen, more micropores and more defects in the metallographic structure, inferior conductivity uniformity, and more frequent abnormal arcing during sputtering. That is, Comparative examples 1 to 3 failed to solve the current technical defects.

The preparation methods of Comparative examples 4 to 18 did not control the D95 particle of the Fe-Mo alloy atomized powder. Therefore, in comparison with the ferrous alloy sputtering targets of Examples 1 to 18, those of

Comparative examples 4 to 18 had the defects of larger Fe-Mo alloy spherical grains, more oxygen amounts, more micropores in the metallographic structure, inferior conductivity uniformity, and more occurrences of abnormal arcing during the sputtering process.

5 To sum up, the present invention controls the composition of the mixed raw materials, the D95 of Fe-Mo alloy atomized powder, and the appropriate sintering temperatures to facilitate solving the sintering issues resulting from the high melting point of molybdenum, thereby obtaining the ferrous alloy sputtering targets with refined spherical grains comprising Fe-Mo alloy.

10 Therefore, the present invention is conducive to alleviate and inhibit the formation of micropores and defects in the metallographic structure, increase the conductivity uniformity of the ferrous alloy sputtering target, lower the occurrence of abnormal arcing during the sputtering process of the ferrous alloy sputtering target, and achieve other beneficial efficacies.

15 The embodiments described above were merely illustrative examples of the present invention and are not intended to limit the scope of the claims herein. The scope of the protection to be claimed is based on the scope of claims, and is not limited to the specific embodiments described above.

WHAT IS CLAIMED IS:

1. A ferrous alloy sputtering target, comprising iron (Fe), molybdenum (Mo) and an additive ingredient, wherein the additive ingredient comprises a first additive metal, a second additive metal or a combination thereof, the first
5 additive metal is selected from the group consisting of titanium (Ti), copper (Cu), germanium (Ge) and combinations thereof, the second additive metal is selected from the group consisting of cobalt (Co), manganese (Mn), silver (Ag), gold (Au), palladium (Pd), platinum (Pt), rhodium (Rh) and combinations thereof; wherein based on a total atom number of the ferrous alloy sputtering
10 target, Fe is in an amount of 25 atomic percent (at%) to 55 at%, and the ferrous alloy sputtering target comprises multiple spherical grains, a composition of the spherical grains comprises a Fe-Mo alloy, and the spherical grains have an average particle size less than 80 microns.

2. The ferrous alloy sputtering target as claimed in Claim 1, wherein the
15 ferrous alloy sputtering target has oxygen in an amount less than 900 parts per million.

3. The ferrous alloy sputtering target as claimed in Claim 1, wherein based on a total atom number of the ferrous alloy sputtering target, the additive ingredient is in an amount of 3 at% to 15 at%.

20 4. The ferrous alloy sputtering target as claimed in Claim 1, wherein based on a total atom number of the ferrous alloy sputtering target, the first additive metal is in an amount of 3 at% to 15 at%.

5. The ferrous alloy sputtering target as claimed in Claim 1, wherein based on a total atom number of the ferrous alloy sputtering target, the second
25 additive metal is in an amount of 3 at% to 15 at%.

6. The ferrous alloy sputtering target as claimed in Claim 1, wherein the

composition of the spherical grains comprises a Fe-Mo-Ti alloy.

7. The ferrous alloy sputtering target as claimed in any one of Claims 1 to 6, wherein the additive ingredient comprises nitride, the nitride comprises titanium nitride, aluminum nitride, boron nitride, chromium nitride, zirconium nitride, tantalum nitride, hafnium nitride, silicon nitride, tungsten nitride, vanadium nitride, titanium carbonitride or combinations thereof.

8. The ferrous alloy sputtering target as claimed in Claim 7, wherein based on a total atom number of the ferrous alloy sputtering target, the nitride is in an amount of 1 at% to 10 at%.

10 9. A preparation method for a ferrous alloy sputtering target, comprising: subjecting an iron raw material and a molybdenum raw material to atomization granulation to obtain a Fe-Mo alloy atomized powder, and the Fe-Mo alloy atomized powder has a D95 less than 125 microns;

mixing the Fe-Mo alloy atomized powder and an additive raw material to
15 obtain a mixed raw material, and the additive raw material comprises a first additive metal raw material, a second additive metal raw material or a combination thereof, the first additive metal raw material is selected from the group consisting of a titanium raw material, a copper raw material, a germanium raw material and combinations thereof, the second additive metal
20 raw material is selected from the group consisting of a cobalt raw material, a manganese raw material, a silver raw material, a gold raw material, a palladium raw material, a platinum raw material, a rhodium raw material and combinations thereof, wherein based on a total atom number of the mixed raw material, the iron raw material is in an amount of 25 at% to 55 at%; and
25 sintering the mixed raw material at a temperature from 1000°C to 1400°C to obtain the ferrous alloy sputtering target;

wherein the ferrous alloy sputtering target comprises multiple spherical grains, a composition of the spherical grains comprises a Fe-Mo alloy, and the spherical grains have an average particle size less than 80 microns.

10. The preparation method as claimed in Claim 9, wherein the ferrous
5 alloy sputtering target has oxygen in an amount less than 900 parts per million.

11. The preparation method as claimed in Claim 9, wherein the additive raw material has a D95 less than 50 microns.

12. The preparation method as claimed in Claim 9, wherein based on a total atom number of the mixed raw material, the additive raw material is in an
10 amount of 3 at% to 15 at%.

13. The preparation method as claimed in Claim 9, wherein the step of subjecting the iron raw material and the molybdenum raw material to atomization granulation to obtain the Fe-Mo alloy atomized powder comprises:
subjecting the iron raw material, the molybdenum raw material, and the
15 titanium raw material to atomization granulation to obtain the Fe-Mo-Ti alloy atomized powder, wherein the Fe-Mo-Ti alloy atomized powder has a D95 less than 125 microns; and

the step of mixing the Fe-Mo alloy atomized powder and the additive raw material to obtain the mixed raw material comprises:

20 mixing the Fe-Mo-Ti alloy atomized powder and the additive raw material to obtain the mixed raw material;

wherein the composition of the spherical grains of the ferrous alloy sputtering target comprises a Fe-Mo-Ti alloy.

14. The preparation method as claimed in any one of Claims 9 to 13,
25 wherein the additive raw material comprises a nitride raw material, the nitride raw material comprises a titanium nitride raw material, an aluminum nitride

raw material, a boron nitride raw material, a chromium nitride raw material, a zirconium nitride raw material, a tantalum nitride raw material, a hafnium nitride raw material, a silicon nitride raw material, a tungsten nitride raw material, a vanadium nitride raw material, a titanium carbonitride raw material
5 or a combination thereof.

15. The preparation method as claimed in Claim 14, wherein based on a total atom number of the mixed raw material, the nitride raw material is in an amount of 1 at% to 10 at%.