

METHOD FOR PREPARING IRON-COBALT-TANTALUM ALLOY POWDER

TECHNICAL FIELD

The present application relates to the technical field of alloy power preparation, for example, to a method for preparing an iron-cobalt-tantalum alloy powder, an iron-cobalt-tantalum alloy powder, and an application thereof.

BACKGROUND

With the rapid development of society and the rapid increase of demand for information storage, recording media using magnetic information storage technology, such as hard disks and optical disks, have been paid more and more attention and occupy a decisive position in the field of information storage due to their advantages of high storage density, large capacity, and low price. Magnetic recording is a way to record information by using magnetic properties, which can input and read information by special methods during storage and use, so as to achieve the purpose of information storage and read. According to the magnetization direction of the medium and the position relationship on the surface of the medium, the magnetic recording includes longitudinal magnetic recording and perpendicular magnetic recording.

At present, the perpendicular magnetic recording technology has completely replaced the longitudinal magnetic recording technology in the magnetic recording market. The perpendicular magnetic recording technology makes the area density and capacity of magnetic recording media increase rapidly, and common magnetic recording media include hard disks, magnetic disks, and optical disks. Therefore, the magnetic recording media generally adopt the design with a multi-layer perpendicular structure, and, taking hard disks as an example, specifically include a lubricating layer, a protective layer, a magnetic recording layer, an intermediate layer, a soft magnetic substrate layer, a substrate layer, and a base layer. The soft magnetic substrate layer mainly functions to record and store data and plays an important role in magnetic recording media. At present, iron-cobalt-tantalum alloy thin films obtained by the sputtering process are the most commonly used soft magnetic substrate layer.

Sputtering is one of the main techniques for preparing thin-film materials and is characterized in that ions generated by an ion source are accelerated and gathered in vacuum to form an ion beam flow with high speed energy, the ion beam flow bombards the surface of a solid so that

kinetic energy exchange occurs between the ions and atoms on the surface of the solid, and the atoms on the surface of the solid are separated from the solid and deposited on the surface of the base. The bombarded solid is a raw material for preparing thin films deposited by a sputtering process, which is generally called sputtering target materials.

5 The iron-cobalt-tantalum alloy sputtering target material used for preparing the soft magnetic substrate layer by the sputtering process needs high relative density, high bending strength, and high magnetic flux. The high relative density can ensure that the thin film obtained by the sputtering process is relatively uniform and avoids abnormal phenomena such as "discharge". The bending strength affects the coercivity of the magnetic material and has a vital impact on
10 the information storage of the magnetic material, and thus, the high bending strength can ensure that the target material is not prone to cracking and other abnormal problems when in use. The target magnetic flux is a very important parameter in magnetic recording target materials, and generally, the higher the target magnetic flux, the stronger the ability of recording and storing data. Therefore, the quality of the iron-cobalt-tantalum alloy sputtering target material is related
15 to the development of soft magnetic substrate layers and even the development of the whole magnetic recording industry.

At present, the existing art discloses some methods for preparing an iron-cobalt-tantalum alloy sputtering target material. For example, CN108004515A discloses a method for preparing an iron-cobalt-tantalum alloy sputtering target material, an iron-cobalt-tantalum alloy sputtering
20 target material and an application thereof. The preparation method includes the following steps: mixing an iron powder, a cobalt powder, and a tantalum powder under the protection of an inert gas to obtain an iron-cobalt-tantalum mixed powder, and then sequentially performing cold pressing, vacuum degassing, primary hot pressing sintering, and secondary hot pressing sintering on the iron-cobalt-tantalum mixed powder to prepare the iron-cobalt-tantalum alloy
25 sputtering target material. However, the preparation method directly uses the mixed powder obtained by mixing an iron powder, a cobalt powder and a tantalum powder according to a required ratio so that only a small amount or almost no compound is formed before the three metals are subjected to the hot pressing sintering, which leads to the magnetism of elemental iron or elemental cobalt and finally leads to the low magnetic flux of the prepared
30 iron-cobalt-tantalum alloy sputtering target material.

CN105473759A discloses a Fe-Co-based alloy sputtering target material, a soft magnetic thin film layer, and a perpendicular magnetic recording medium produced using the soft magnetic thin film layer. The preparation method includes the steps of preparing a Fe-Co-based alloy

powder and sintering the powder under pressure. The preparation method directly uses the Fe-Co-based alloy powder obtained by gas atomization, and the prepared Fe-Co-based alloy sputtering target material can meet the properties of high purity, high density, high bending strength, high magnetic flux, and the like and meet the requirements of magnetron sputtering.

5 However, the melt liquid of Fe-Co-based alloy is very viscous and can easily block the pipeline, resulting in the failure of gas atomization. In addition, there is no Fe-Co-based alloy powder that meets the target ratio requirement to purchase in the market, which greatly hinders the preparation and research and development of the Fe-Co-based alloy sputtering target material.

10 To sum up, it is urgent to develop a method for preparing an iron-cobalt-tantalum alloy powder, an iron-cobalt-tantalum alloy powder, and an application thereof.

SUMMARY

15 The present application provides a method for preparing an iron-cobalt-tantalum alloy powder, an iron-cobalt-tantalum alloy powder, and an application thereof. The preparation method controls the melting temperature of the iron-cobalt-tantalum mixture to be 1650°C to 1750°C and optimizes the atomization conditions so that the prepared iron-cobalt-tantalum alloy powder has a purity of greater than or equal to 99.95% and oxygen content of less than or equal to 600 ppm. The iron-cobalt-tantalum alloy powder with a particle size of less than 150 mesh obtained by sieving facilitates the preparation of the iron-cobalt-tantalum alloy sputtering target material with a high purity, high density, high bending strength, and qualified magnetic flux.

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

A first object of the present application is to provide a method for preparing an iron-cobalt-tantalum alloy powder. The preparation method includes the following steps:

(1) preparing and mixing an iron material, a cobalt material, and a tantalum material according to a target atomic ratio to obtain an iron-cobalt-tantalum mixture;

25 (2) completely melting the iron-cobalt-tantalum mixture obtained in step (1) at a temperature of 1650°C to 1750°C to obtain an iron-cobalt-tantalum melt; and

(3) atomizing the iron-cobalt-tantalum melt obtained in step (2) under the action of an inert gas to obtain the iron-cobalt-tantalum alloy powder.

The preparation method provided by the present application controls the melting temperature of the iron-cobalt-tantalum mixture to be 1650°C to 1750°C and optimizes the atomization conditions, avoiding the problem that the iron-cobalt-tantalum melt blocks the pipeline due to viscosity, so that the prepared iron-cobalt-tantalum alloy powder has a purity of greater than or equal to 99.95% and oxygen content of less than or equal to 600 ppm. The iron-cobalt-tantalum alloy powder with a particle size of less than 150 mesh obtained by sieving facilitates the preparation of the iron-cobalt-tantalum alloy sputtering target material with high purity, high density, high bending strength, and qualified magnetic flux.

In the present application, the iron-cobalt-tantalum mixture is melted at a temperature of 1650°C to 1750°C, for example, 1650°C, 1670°C, 1690°C, 1700°C, 1710°C, 1730°C, or 1750°C, etc. However, the temperature is not limited to the values listed above, and other unlisted values within this value range are also applicable.

As an optional technical solution of the present application, in the target atomic ratio in step (1), the atomic ratio of cobalt is 28.5% to 29.5%, the atomic ratio of tantalum is 17.5% to 18.5%, and the remainder is iron and unavoidable impurities.

In the present application, the atomic ratio of cobalt is 28.5% to 29.5%, for example, 28.5%, 28.8%, 29%, 29.2%, or 29.5%, etc. However, the atomic ratio of cobalt is not limited to the values listed above, and other unlisted values within this value range are also applicable.

In the present application, the atomic ratio of tantalum is 17.5% to 18.5%, for example, 17.5%, 17.7%, 18%, 18.3%, or 18.5%, etc. However, the atomic ratio of tantalum is not limited to the values listed above, and other unlisted values within this value range are also applicable.

In the present application, the total content of the unavoidable impurities is less than 0.05%, ensuring that the purity of the prepared iron-cobalt-tantalum alloy powder is greater than or equal to 99.95%.

As an optional technical solution of the present application, the iron material in step (1) has a purity of greater than or equal to 99.95%.

Optionally, the iron material in step (1) is an electrolytic iron material.

Optionally, the cobalt material in step (1) has a purity of greater than or equal to 99.95%.

Optionally, the cobalt material in step (1) is an electrolytic cobalt material.

Optionally, the tantalum material in step (1) has a purity of greater than or equal to 99.95%.

Optionally, the tantalum material in step (1) is a tantalum scrap of a tantalum target material.

5 The tantalum target material in the present application may be the tantalum target material disclosed in CN102517531A, CN103572223A or CN103572225A. In the process of machining the tantalum target material to prepare the target material assembly, a large number of tantalum scraps of the tantalum target material are generated.

10 As an optional technical solution of the present application, before the iron material, the cobalt material, and the tantalum material are prepared according to the target atomic ratio in step (1), the method further includes the step of pretreating the iron material, the cobalt material, and the tantalum material respectively.

Optionally, the pretreatment sequentially includes deoiling, acid pickling, drying, and vacuum packaging.

15 As is well known to those skilled in the art, in the process from the preparation of powder preparation materials to the powder preparation, the powder preparation materials are also subjected to the processes such as transportation and storage. Therefore, it is necessary to perform vacuum packaging on the pretreated powder preparation materials, which facilitates the isolation of the materials from dust and air, reduces the oxygen absorption reaction of the powder preparation materials, and further ensures that the finally prepared iron-cobalt-tantalum
20 alloy powder meets the requirement of the oxygen content of less than or equal to 600 ppm. In addition, the materials can be used by just open the vacuum packages when the powder is officially prepared.

25 Optionally, the deoiling includes the following steps: putting the iron material, the cobalt material, and the tantalum material into a cleaning liquid respectively for ultrasonic cleaning, then washing the iron material, the cobalt material, and the tantalum material with pure water, and wiping the iron material, the cobalt material, and the tantalum material clean.

Optionally, the cleaning liquid is an aqueous solution containing a detergent.

In the present application, there is no concentration limitation to the aqueous solution containing

a detergent, and those skilled in the art can reasonably select the type and concentration of the detergent according to the actual situation, as long as the oil stain can be cleaned.

5 Optionally, the ultrasonic cleaning is performed for 50 minutes to 70 minutes, for example, 50 minutes, 52 minutes, 55 minutes, 57 minutes, 60 minutes, 63 minutes, 65 minutes, 68 minutes, or 70 minutes, etc. However, the time is not limited to the values listed above, and other unlisted values within this value range are also applicable.

10 Optionally, the ultrasonic cleaning is performed at a temperature of 50°C to 70°C, for example, 50°C, 52°C, 55°C, 57°C, 60°C, 63°C, 65°C, 68°C, or 70°C, etc. However, the temperature is not limited to the values listed above, and other unlisted values within this value range are also applicable.

Optionally, the acid pickling includes the following steps: putting the iron material, the cobalt material, and the tantalum material into a pickling solution for ultrasonic cleaning, then washing the iron material, the cobalt material, and the tantalum material with pure water, and wiping the iron material, the cobalt material, and the tantalum material clean

15 Optionally, the pickling solution is an aqueous solution of nitric acid and hydrofluoric acid.

Optionally, the nitric acid and hydrofluoric acid in the pickling solution are 38.5% and 7.1% respectively, based on percentage by mass.

20 Optionally, the acid pickling is performed for 10 minutes to 30 minutes, for example, 10 minutes, 13 minutes, 15 minutes, 17 minutes, 20 minutes, 23 minutes, 25 minutes, 27 minutes, or 30 minutes, etc. However, the time is not limited to the values listed above, and other unlisted values within this value range are also applicable.

Optionally, the drying is performed in a vacuum-drying oven.

25 Optionally, the drying is performed for 60 minutes to 90 minutes, for example, 60 minutes, 65 minutes, 70 minutes, 75 minutes, 80 minutes, 85 minutes, or 90 minutes, etc. However, the time is not limited to the values listed above, and other unlisted values within this value range are also applicable.

Optionally, the drying is performed at a temperature of 65°C to 80°C, for example, 65°C, 67°C, 69°C, 70°C, 72°C, 74°C, or 75°C, etc. However, the temperature is not limited to the values

listed above, and other unlisted values within this value range are also applicable.

In the present application, the pretreatment sequentially includes deoiling, acid pickling, drying, and vacuum packaging, which can not only remove impurities such as oil stains, dust, and water stains on the surfaces of the iron material, cobalt material, and tantalum material, but also effectively prevent the iron material, cobalt material, and tantalum material from being oxidized by air, thereby ensuring the purity requirement of the prepared iron-cobalt-tantalum alloy powder.

As an optional technical solution of the present application, the mixing in step (1) is performed in a melting crucible in a manner of layering and superposition, which specifically includes the following steps:

paving the cobalt material on the bottom of the melting crucible, paving the tantalum material on the cobalt material, and paving the iron material on the tantalum material to form a cobalt-tantalum-iron material-paving unit, and repeating the above operations according to the cobalt-tantalum-iron material-paving unit until the loading is completed.

In the present application, materials are mixed based on the cobalt-tantalum-iron material-paving unit according to the manner of layering and superposition, which can avoid local aggregation of tantalum materials with high melting point and large viscosity and prevent the problems that the composition of the subsequently obtained iron-cobalt-tantalum melt are uneven and the iron-cobalt-tantalum melt blocks the pipeline due to excessive local viscosity.

As an optional technical solution of the present application, the melting in step (2) is performed at an absolute vacuum degree of less than or equal to 10 Pa.

Optionally, the melting in step (2) is performed in a vacuum melting chamber, and then the iron-cobalt-tantalum melt is poured into a tundish. The tundish is controlled to be at a temperature of 1550°C to 1650°C, for example, 1550°C, 1560°C, 1580°C, 1600°C, 1610°C, 1630°C, or 1650°C, etc. However, the temperature is not limited to the values listed above, and other unlisted values within this value range are also applicable.

In the present application, the melting is controlled to be performed at a temperature of 1650°C to 1750°C and an absolute vacuum degree of less than or equal to 10 Pa, which can fully ensure that the iron-cobalt-tantalum mixture can be completely melted and mixed evenly. Further, the

obtained iron-cobalt-tantalum melt is poured into a tundish connected with an atomizing nozzle, and the temperature is controlled to be 1550°C to 1650°C, which can effectively prevent the iron-cobalt-tantalum melt from blocking the tundish and effectively prevent the iron-cobalt-tantalum melt from blocking the flow guide pipe between the tundish and the atomizing nozzle.

As an optional technical solution of the present application, the inert gas in step (3) is argon gas.

Optionally, the argon gas has a purity of greater than or equal to 5N.

Optionally, the atomizing in step (3) is performed at a pressure of 3 MPa to 4 MPa, for example, 3 MPa, 3.2 MPa, 3.4 MPa, 3.5 MPa, 3.7 MPa, 3.9 MPa, or 4 MPa, etc. However, the pressure is not limited to the values listed above, and other unlisted values within this value range are also applicable.

In the present application, the pressure of the atomizing is set based on the high viscosity of the iron-cobalt-tantalum melt and needs to be strictly controlled in the range of 3 MPa to 4 MPa, which can prevent the downward flow of the iron-cobalt-tantalum melt from being hindered due to a large atomization pressure and can also prevent the average particle of the iron-cobalt-tantalum alloy powder from being large due to a small atomization pressure, thereby preventing the low yield of the subsequent iron-cobalt-tantalum alloy sputtering target material.

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

(1) preparing the pretreated iron material, cobalt material, and tantalum material according to a target atomic ratio, and mixing the iron material, the cobalt material, and the tantalum material in a melting crucible in a manner of layering and superposition, where step (1) specifically includes the following steps: paving the cobalt material on the bottom of the melting crucible, paving the tantalum material on the cobalt material, paving the iron material on the tantalum material to form a cobalt-tantalum-iron material-paving unit, and repeating the above operations according to the cobalt-tantalum-iron material-paving unit until the loading is completed, so as to obtain the iron-cobalt-tantalum mixture;

wherein in the target atomic ratio, the atomic ratio of cobalt is 28.5% to 29.5%, the atomic ratio of tantalum is 17.5% to 18.5%, and the remainder is iron and unavoidable impurities;

the iron material has a purity of greater than or equal to 99.95%, the cobalt material has a purity of greater than or equal to 99.95%, and the tantalum material has a purity of greater than or equal to 99.95%; and

the pretreatment sequentially includes deoiling, acid pickling, drying, and vacuum packaging;

5 (2) putting the iron-cobalt-tantalum mixture obtained in step (1) into a vacuum melting chamber with an absolute vacuum degree controlled to be less than or equal to 10 Pa, and completely melting the iron-cobalt-tantalum mixture at 1650°C to 1750°C to obtain an iron-cobalt-tantalum melt; and

10 (3) pouring the iron-cobalt-tantalum melt obtained in step (2) into a tundish whose temperature is controlled to be 1550°C to 1650°C, and performing atomization under the action of argon gas with a purity of greater than or equal to 5N at a pressure controlled to be 3 MPa to 4 MPa, so as to obtain the iron-cobalt-tantalum alloy powder.

15 In the present application, the preparation method belongs to the gas atomization powder preparation method in which a metal liquid or alloy liquid is crushed into fine droplets through the impingement effect of a fast-moving, high-pressure gas which serves as an atomizing medium, and then the fine droplets are condensed into solid powder. The gas atomization method is the best method to produce fully alloyed powders. Each particle of the prepared alloy powder has the same uniform chemical composition as the given molten alloy, and the crystalline structure is refined due to rapid solidification, which eliminates the macrosegregation
20 of the second phase.

A second object of the present application is to provide an iron-cobalt-tantalum alloy powder which is prepared by using the preparation method described in the first object.

25 A third object of the present application is to provide an application of the iron-cobalt-tantalum alloy powder. The iron-cobalt-tantalum alloy powder described in the second object is sieved, and the iron-cobalt-tantalum alloy powder with a particle size of less than 150 mesh is used for preparing an iron-cobalt-tantalum alloy sputtering target material and especially, used in the process of preparing an iron-cobalt-tantalum alloy sputtering target material by hot isostatic pressing.

Compared with the existing art, the present application at least has beneficial effects described

below.

(1) The preparation method provided by the present application controls the melting temperature of the iron-cobalt-tantalum mixture to be 1650°C to 1750°C and optimizes the atomization conditions so that the prepared iron-cobalt-tantalum alloy powder has a purity of greater than or equal to 99.95% and oxygen content of less than or equal to 600 ppm. The iron-cobalt-tantalum alloy powder with a particle size of less than 150 mesh obtained by sieving facilitates the preparation of the iron-cobalt-tantalum alloy sputtering target material with high purity, high density, high bending strength, and qualified magnetic flux.

(2) The preparation method provided by the present application uses the tantalum scraps of the tantalum target material as the tantalum material, which not only solves the treatment problem of the tantalum scraps generated in the machining process of the tantalum target material but also can prepare the iron-cobalt-tantalum alloy powder with extremely high application value, thereby turning waste into wealth.

BRIEF DESCRIPTION OF DRAWINGS

FIG. 1 is a scanning electron microscope image of an iron-cobalt-tantalum alloy powder according to Example 1 of the present application; and

FIG. 2 is an energy dispersive spectrometry (EDS) graph of an iron-cobalt-tantalum alloy powder according to Example 1 of the present application.

DETAILED DESCRIPTION

The technical solutions of the present application are further described below through embodiments in conjunction with drawings. Those skilled in the art are to understand that examples described herein are merely used for a better understanding of the present application and are not to be construed as specific limitations to the present application.

Example 1

This example provides a method for preparing an iron-cobalt-tantalum alloy powder. The preparation method includes the following steps.

(1) An electrolytic iron material with a purity of greater than or equal to 99.95%, electrolytic

cobalt material with a purity of greater than or equal to 99.95%, and tantalum scraps of a tantalum target material disclosed in CN102517531A (with a purity of greater than or equal to 99.95%) were sequentially subjected to the pretreatment of deoiling, acid pickling, drying, and vacuum packaging.

- 5 First, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were respectively put into an aqueous solution containing a detergent, subjected to ultrasonic cleaning at 60°C for 60 minutes for deoiling, then washed with pure water, and finally wiped clean. Then the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were subjected to ultrasonic cleaning in a pickling solution for 20 minutes for acid pickling, where
10 the nitric acid and hydrofluoric acid in the pickling solution were 38.5% and 7.1% respectively, based on percentage by mass, then washed with pure water, and finally wiped clean. After that, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps obtained after pickling were respectively put into a vacuum-drying oven and dried at 70°C for 80 minutes. Finally, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps
15 were subjected to vacuum packaging for standby.

The pretreated electrolytic iron material, electrolytic cobalt material, and tantalum scraps were prepared according to a target atomic ratio in which the atomic ratio of cobalt was 29%, the atomic ratio of tantalum was 18%, and the remainder was iron and unavoidable impurities and then mixed in a melting crucible in a manner of layering and superposition. The operation of
20 mixing specifically includes the following steps: the cobalt material was paved on the bottom of the melting crucible, the tantalum material was paved on the cobalt material, then the iron material was paved on the tantalum material to form a cobalt-tantalum-iron material-paving unit, and then the above operations were repeated according to the cobalt-tantalum-iron material-paving unit until the loading was completed, so as to obtain the iron-cobalt-tantalum
25 mixture.

(2) The iron-cobalt-tantalum mixture obtained in step (1) was put into a vacuum melting chamber with an absolute vacuum degree controlled to be less than or equal to 10 Pa and completely melted at 1700°C to obtain an iron-cobalt-tantalum melt.

(3) The iron-cobalt-tantalum melt obtained in step (2) was poured into a tundish whose
30 temperature was controlled to be 1600°C and atomized under the action of argon gas with a purity of greater than or equal to 5N at a pressure controlled to be 3.5 MPa, so as to obtain the iron-cobalt-tantalum alloy powder.

The scanning electron microscopy-energy dispersive spectrometry (SEM-EDS) detection is performed on the iron-cobalt-tantalum alloy powder in this example. The scanning electron microscope image is shown in FIG. 1, the EDS energy spectrum graph is shown in FIG. 2, and the EDS energy spectrum detection results are summarized in Table 1. Wherein, as can be seen from FIG. 1, the sphericity of the iron-cobalt-tantalum alloy powder is relatively high, and as can be seen from Table 1, the composition of the iron-cobalt-tantalum alloy powder meets the target atomic ratio.

Table 1

Element	Mass percent/%	atomic percent/%	Error/%
Fe	37.04	52.52	1.43
Co	21.98	29.54	1.14
Ta	40.98	17.94	1.91
Total	100.00	100.00	/

Example 2

- 10 This example provides a method for preparing an iron-cobalt-tantalum alloy powder, which is the same as the preparation method in Example 1 except that "an absolute vacuum degree controlled to be less than or equal to 10 Pa" in step (2) was replaced with "an absolute vacuum degree controlled to fluctuate between 12 Pa to 15 Pa".

Example 3

- 15 This example provides a method for preparing an iron-cobalt-tantalum alloy powder, which is the same as the preparation method in Example 1 except that "a pressure controlled to be 3.5 MPa" in step (3) was replaced with "a pressure controlled to be 2.5 MPa".

Example 4

- 20 This example provides a method for preparing an iron-cobalt-tantalum alloy powder, which is the same as the preparation method in Example 1 except that "a pressure controlled to be 3.5 MPa" in step (3) was replaced with "a pressure controlled to be 4.5 MPa".

Example 5

This example provides a method for preparing an iron-cobalt-tantalum alloy powder. The

preparation method includes the following steps.

(1) An electrolytic iron material with a purity of greater than or equal to 99.95%, electrolytic cobalt material with a purity of greater than or equal to 99.95%, and tantalum scraps of a tantalum target material disclosed in CN102517531A (with a purity of greater than or equal to 99.95%) were sequentially subjected to the pretreatment of deoiling, acid pickling, drying, and vacuum packaging.

First, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were respectively put into an aqueous solution containing a detergent, subjected to ultrasonic cleaning at 50°C for 70 minutes for deoiling, then washed with pure water, and finally wiped clean. Then the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were subjected to ultrasonic cleaning in a pickling solution for 10 minutes for acid pickling, where the nitric acid and hydrofluoric acid in the pickling solution were 38.5% and 7.1% respectively, based on percentage by mass, then washed with pure water, and finally wiped clean. After that, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps obtained after pickling were respectively put into a vacuum-drying oven and dried at 65°C for 90 minutes. Finally, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were subjected to vacuum packaging for standby.

The pretreated electrolytic iron material, electrolytic cobalt material, and tantalum scraps were prepared according to a target atomic ratio in which the atomic ratio of cobalt was 28.5%, the atomic ratio of tantalum was 18.5%, and the remainder was iron and unavoidable impurities and then mixed in a melting crucible in a manner of layering and superposition. The operation of mixing specifically includes the following steps: the cobalt material was paved on the bottom of the melting crucible, the tantalum material was paved on the cobalt material, then the iron material was paved on the tantalum material to form a cobalt-tantalum-iron material-paving unit, and then the above operations were repeated according to the cobalt-tantalum-iron material-paving unit until the loading was completed, so as to obtain the iron-cobalt-tantalum mixture.

(2) The iron-cobalt-tantalum mixture obtained in step (1) was put into a vacuum melting chamber with an absolute vacuum degree controlled to be less than or equal to 10 Pa and completely melted at 1750°C to obtain an iron-cobalt-tantalum melt.

(3) The iron-cobalt-tantalum melt obtained in step (2) was poured into a tundish whose

temperature was controlled to be 1650°C and atomized under the action of argon gas with a purity of greater than or equal to 5N at a pressure controlled to be 4 MPa, so as to obtain the iron-cobalt-tantalum alloy powder.

Example 6

- 5 This example provides a method for preparing an iron-cobalt-tantalum alloy powder. The preparation method includes the following steps.

(1) An electrolytic iron material with a purity of greater than or equal to 99.95%, electrolytic cobalt material with a purity of greater than or equal to 99.95%, and tantalum scraps of a tantalum target material disclosed in CN102517531A (with a purity of greater than or equal to
10 99.95%) were sequentially subjected to the pretreatment of deoiling, acid pickling, drying, and vacuum packaging.

First, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were respectively put into an aqueous solution containing a detergent, subjected to ultrasonic cleaning at 70°C for 50 minutes for deoiling, then washed with pure water, and finally wiped clean. Then
15 the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were subjected to ultrasonic cleaning in a pickling solution for 30 minutes for acid pickling, where the nitric acid and hydrofluoric acid in the pickling solution were 38.5% and 7.1% respectively, based on percentage by mass, then washed with pure water, and finally wiped clean. After that, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps obtained
20 after pickling were respectively put into a vacuum-drying oven and dried at 75°C for 60 minutes. Finally, the electrolytic iron material, the electrolytic cobalt material, and the tantalum scraps were subjected to vacuum packaging for standby.

The pretreated electrolytic iron material, electrolytic cobalt material, and tantalum scraps were prepared according to a target atomic ratio in which the atomic ratio of cobalt was 29.5%, the
25 atomic ratio of tantalum was 17.5%, and the remainder was iron and unavoidable impurities and then mixed in a melting crucible in a manner of layering and superposition. The operation of mixing specifically includes the following steps: the cobalt material was paved on the bottom of the melting crucible, the tantalum material was paved on the cobalt material, then the iron material was paved on the tantalum material to form a cobalt-tantalum-iron material-paving unit,
30 and then the above operations were repeated according to the cobalt-tantalum-iron material-paving unit until the loading was completed, so as to obtain the iron-cobalt-tantalum

mixture.

(2) The iron-cobalt-tantalum mixture obtained in step (1) was put into a vacuum melting chamber with an absolute vacuum degree controlled to be less than or equal to 10 Pa and completely melted at 1650°C to obtain an iron-cobalt-tantalum melt.

- 5 (3) The iron-cobalt-tantalum melt obtained in step (2) was poured into a tundish whose temperature was controlled to be 1550°C and atomized under the action of argon gas with a purity of greater than or equal to 5N at a pressure controlled to be 3 MPa, so as to obtain the iron-cobalt-tantalum alloy powder.

Comparative Example 1

- 10 This comparative example provides a method for preparing an iron-cobalt-tantalum alloy powder, which is the same as the preparation method in Example 1 except that "the iron-cobalt-tantalum mixture obtained was completely melted at 1700°C" in step (2) was replaced with "the iron-cobalt-tantalum mixture obtained was completely melted at 1600°C".

- 15 The iron-cobalt-tantalum alloy powders prepared in the above examples and comparative example were tested in the following aspects:

sphericity: determined according to the scanning electron microscope image obtained by the SEM-EDS detection;

- 20 oxygen content: tested according to the thermal conductivity method disclosed in the national standard GB/T 14265-2017 *General rule of chemical analysis for hydrogen, oxygen, nitrogen, carbon and sulfur in metallic materials*; and

finished product rate: the mass percentage of iron-cobalt-tantalum alloy powder with a particle size of less than 150 mesh obtained by sieving with respect to the feeding quantity of iron material, cobalt material and tantalum material.

- 25 The related test results of the iron-cobalt-tantalum alloy powders prepared in the above examples and comparative example are shown in Table 2.

Table 2

Term	Sphericity	Oxygen content	Finished product rate
Example 1	Good	451 ppm	81%
Example 2	Good	568 ppm	80%
Example 3	Good	472 ppm	60%
Example 4	Good	461 ppm	35%
Example 5	Good	486 ppm	82%
Example 6	Good	501 ppm	80%
Comparative Example 1	Good	660 ppm	70%

The following conclusions can be obtained from Table 2.

(1) The preparation method provided by the present application controls the melting temperature of the iron-cobalt-tantalum mixture to be 1650°C to 1750°C and optimizes the atomization conditions so that the prepared iron-cobalt-tantalum alloy powder has a good sphericity and oxygen content of less than or equal to 600 ppm. The iron-cobalt-tantalum alloy powder with a particle size of less than 150 mesh obtained by sieving has a high finished product rate which can reach 82%, which facilitates the preparation of the iron-cobalt-tantalum alloy sputtering target material with high purity, high density, high bending strength, and qualified magnetic flux. In addition, the tantalum scraps of the tantalum target material serve as the tantalum material, which turning waste into wealth.

(2) As can be seen from the comparison between Example 1 and Example 2, in Example 2, in the process of melting the iron-cobalt-tantalum mixture, the absolute vacuum degree is controlled to fluctuate between 12 Pa and 15 Pa, resulting in the oxygen content of the prepared iron-cobalt-tantalum alloy powder of 568 ppm.

(3) As can be seen from the comparison between Example 1 and Examples 3 and 4, the pressure in the gas atomization process in Example 3 is controlled to be lower than 3 to 4 MPa, which is only 2.5 MPa, resulting in the large average particle size of the prepared iron-cobalt-tantalum alloy powder and the decrease of the finished product rate to 60%; and the pressure in the gas atomization process in Example 4 is controlled to exceed 3 to 4 MPa, which reaches up to 4.5 MPa, resulting in the large downward flow resistance of the iron-cobalt-tantalum melt and the finished product rate of the prepared iron-cobalt-tantalum alloy powder of only 35%.

(4) As can be seen from the comparison between Example 1 and Comparative Example 1, the melting temperature of the iron-cobalt-tantalum mixture in Comparative Example 1 is lower than 1650-1750°C, which is only 1600°C, resulting in the problem of the uneven composition of the iron-cobalt-tantalum melt so that the oxygen content of the prepared iron-cobalt-tantalum alloy powder is as high as 660 ppm.

The applicant has stated that although the detailed structure characteristics of the present application are described through the examples described above, the present application is not limited to the detailed structure characteristics described above, which means that the implementation of the present application does not necessarily depend on the detailed structure characteristics described above. It should be apparent to those skilled in the art that any improvements made to the present application, equivalent replacements of units selected in the present application and addition of auxiliary units thereof, and selections of specific methods, etc., all fall within the protection scope and the disclosed scope of the present application.

Though the optional embodiments of the present application have been described above in detail, the present application is not limited to details of the above-described embodiments, and various simple modifications can be made to the technical solutions of the present application without departing from the scope of the present application. These simple modifications are all within the protection scope of the present application.

In addition, it is to be noted that if not in collision, the specific technical features described in the above specific embodiments may be combined in any suitable manner. In order to avoid unnecessary repetition, the present application does not further specify any of various possible combination manners.

In addition, the various examples of the present application may be combined arbitrarily without departing from the idea of the present application, and these combinations are likewise to be considered as the disclosure of the present application.

What is claimed is:

1. A method for preparing an iron-cobalt-tantalum alloy powder, comprising:

(1) preparing and mixing an iron material, a cobalt material, and a tantalum material according to a target atomic ratio to obtain an iron-cobalt-tantalum mixture;

5 (2) completely melting the iron-cobalt-tantalum mixture obtained in step (1) at a temperature of 1650°C to 1750°C to obtain an iron-cobalt-tantalum melt; and

(3) atomizing the iron-cobalt-tantalum melt obtained in step (2) under the action of an inert gas to obtain the iron-cobalt-tantalum alloy powder;

10 wherein the mixing in step (1) is performed in a melting crucible in a manner of layering and superposition, which specifically comprises:

paving the cobalt material on the bottom of the melting crucible, paving the tantalum material on the cobalt material, and paving the iron material on the tantalum material to form a cobalt-tantalum-iron material-paving unit, and repeating the above operations according to the cobalt-tantalum-iron material-paving unit until the loading is completed.

15 2. The preparation method according to claim 1, wherein in the target atomic ratio in step (1), the atomic ratio of cobalt is 28.5% to 29.5%, the atomic ratio of tantalum is 17.5% to 18.5%, and the remainder is iron and unavoidable impurities.

3. The preparation method according to claim 1 or 2, wherein the iron material in step (1) has a purity of greater than or equal to 99.95%.

20 4. The preparation method according to any one of claims 1 to 3, wherein the iron material in step (1) is an electrolytic iron material;

optionally, the cobalt material in step (1) has a purity of greater than or equal to 99.95%;

optionally, the cobalt material in step (1) is an electrolytic cobalt material;

optionally, the tantalum material in step (1) has a purity of greater than or equal to 99.95%;

25 and

optionally, the tantalum material in step (1) is a tantalum scrap of a tantalum target material.

5. The preparation method according to any one of claims 1 to 4, wherein, before preparing the iron material, the cobalt material, and the tantalum material according to the target atomic ratio in step (1), further comprising: pretreating the iron material, the cobalt material, and the

tantalum material respectively;

optionally, the pretreatment sequentially comprises deoiling, acid pickling, drying, and vacuum packaging;

5 optionally, the deoiling comprises the following steps: putting the iron material, the cobalt material, and the tantalum material into a cleaning liquid respectively for ultrasonic cleaning, washing the iron material, the cobalt material, and the tantalum material with pure water, and wiping the iron material, the cobalt material, and the tantalum material clean;

optionally, the cleaning liquid is an aqueous solution containing a detergent;

optionally, the ultrasonic cleaning is performed for 50 minutes to 70 minutes;

10 optionally, the ultrasonic cleaning is performed at a temperature of 50°C to 70°C;

optionally, the acid pickling comprises the following steps: putting the iron material, the cobalt material, and the tantalum material into a pickling solution for ultrasonic cleaning, washing the iron material, the cobalt material, and the tantalum material with pure water, and wiping the iron material, the cobalt material, and the tantalum material clean;

15 optionally, the pickling solution is an aqueous solution of nitric acid and hydrofluoric acid;

optionally, the nitric acid and hydrofluoric acid in the pickling solution are 38.5% and 7.1% respectively, based on percentage by mass;

optionally, the acid pickling is performed for 10 minutes to 30 minutes;

optionally, the drying is performed in a vacuum-drying oven;

20 optionally, the drying is performed for 60 minutes to 90 minutes; and

optionally, the drying is performed at a temperature of 65°C to 80°C.

6. The preparation method according to any one of claims 1 to 5, wherein the melting in step (2) is performed at an absolute vacuum degree of less than or equal to 10 Pa; and

25 optionally, the melting in step (2) is performed in a vacuum melting chamber, and then the iron-cobalt-tantalum melt is poured into a tundish, wherein the tundish is controlled to be at a temperature of 1550°C to 1650°C.

7. The preparation method according to any one of claims 1 to 6, wherein the inert gas in step (3) is argon gas;

optionally, the argon gas has a purity of greater than or equal to 5N; and

optionally, the atomizing in step (3) is performed at a pressure of 3 MPa to 4 MPa.

8. The preparation method according to any one of claims 1 to 7, comprising the following steps:

- 5 (1) preparing the pretreated iron material, cobalt material, and tantalum material according to a target atomic ratio, and mixing the iron material, the cobalt material, and the tantalum material in a melting crucible in a manner of layering and superposition, wherein step (1) specifically comprises the following steps: paving the cobalt material on the bottom of the melting crucible, paving the tantalum material on the cobalt material, paving the iron material
10 on the tantalum material to form a cobalt-tantalum-iron material-paving unit, and repeating the above operations according to the cobalt-tantalum-iron material-paving unit until the loading is completed, so as to obtain the iron-cobalt-tantalum mixture;

wherein in the target atomic ratio, the atomic ratio of cobalt is 28.5% to 29.5%, the atomic ratio of tantalum is 17.5% to 18.5%, and the remainder is iron and unavoidable impurities;

- 15 the iron material has a purity of greater than or equal to 99.95%, the cobalt material has a purity of greater than or equal to 99.95%, and the tantalum material has a purity of greater than or equal to 99.95%; and

the pretreatment sequentially comprises deoiling, acid pickling, drying, and vacuum packaging;

- 20 (2) putting the iron-cobalt-tantalum mixture obtained in step (1) into a vacuum melting chamber with an absolute vacuum degree controlled to be less than or equal to 10 Pa, and completely melting the iron-cobalt-tantalum mixture at 1650°C to 1750°C to obtain an iron-cobalt-tantalum melt; and

- 25 (3) pouring the iron-cobalt-tantalum melt obtained in step (2) into a tundish whose temperature is controlled to be 1550°C to 1650°C, and performing atomization under the action of argon gas with a purity of greater than or equal to 5N at a pressure controlled to be 3 MPa to 4 MPa, so as to obtain the iron-cobalt-tantalum alloy powder.