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(54) METHOD FOR NITRIDING RARE-EARTH MAGNET, AND NITRIDED RARE-EARTH MAGNET

(57) A method for nitriding a rare-earth magnet, comprising: 1) vacuumizing, adding a nitrogen source into a reactor filled with the rare-earth magnet, and performing three-stage heating treatment to obtain a semi-finished product, the heating temperature of the three-stage heating treatment is increased stage by stage; and 2) raising the temperature of the semi-finished product to a first set temperature, then adjusting the temperature to a second set temperature, vacuumizing, introducing inert gas, and performing heat preservation at the second set temperature to obtain a nitrided rare-earth magnet, the second set temperature being lower than the first set temperature.

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Description

TECHNICAL FIELD

[0001] The present application belongs to the field of magnetic materials, and relates to a method for nitriding a rare-earth magnet, and a nitrided rare-earth magnet.

BACKGROUND

[0002] In recent years, with the advancement and development of science and technology, the application scope of magnetic materials is more and more wide, and people's life is increasingly inseparable from magnetic materials. Magnetic materials are divided into permanent magnet materials and soft magnetic materials, and different properties have different uses. Rare-earth magnetic materials are widely used due to their excellent magnetic properties.

[0003] The most commonly used rare-earth permanent magnet material is NdFeB, which is currently a rare-earth permanent magnet material with the highest magnetic energy product of permanent magnetic properties. However, due to the low content of rare-earth elements in the earth crust and the high cost of mining and purification, NdFeB materials have high performance and high cost.

[0004] Samarium-iron-nitrogen magnets avoid the shortcomings of neodymium-iron-boron magnets, such as low Curie temperature, easy oxidation and high cost, and become a research hotspot in the new generation of rare-earth permanent magnet materials. The introduction of nitrogen atoms does not change the crystal structure of samarium ferroalloy, but causes lattice expansion, which enhances the ferromagnetic coupling exchange effect of the alloy, greatly improves the Curie temperature of the alloy, and enhances the anisotropy field. It is the key to improve the permanent magnetic properties of samarium ferroalloy, so that the nitrogen treatment process plays a vital role in the preparation of samarium-iron-nitrogen magnets. At present, the relatively mature nitriding process at home and abroad is to obtain alloy powder by mechanical method, and then subject to nitrogen treatment. There are some problems in solid powder nitriding, such as nitriding nonuniform, incomplete, low efficiency and easy oxidation of powder.

[0005] There are rare-earth-iron-nitrogen materials, including SmFeN, NdFeN, CeFeN, etc., some use the permanent magnet properties of materials, and some use the soft magnetic properties of materials. However, in the preparation process, both of the preparation methods are forming a rare-earth iron alloy first, and then subjecting the alloy to nitriding.

[0006] The computer, mobile phone and network equipment used in modern communication will produce electromagnetic interference and electromagnetic radiation during use. In order to effectively reduce and eliminate electromagnetic interference and radiation, it is necessary to use wave-absorbing materials with high complex permeability. At present, ferrite materials widely used have the disadvantages of narrow frequency band and low complex permeability at high frequency. Moreover, metal soft magnetic materials have eddy current loss, resulting in low high frequency soft magnetic performance. CeFeN and other materials can maintain high permeability and wide resonance frequency at high frequency, realize electromagnetic shielding and reduce signal noise in a wide frequency band, and meet the requirements of modern technology, which is widely used in instruments, meters, communications and other fields.

[0007] Rare-earth transition metal magnetic materials show excellent magnetic properties after nitrogen treatment, but insufficient nitrogen content and uneven distribution of nitrogen elements in the nitriding process seriously affect the performance of the materials.

[0008] CN101699578A discloses a high frequency soft magnetic material composed of rare-earth, iron and nitrogen, a composite material prepared by the same and a preparation method thereof. Firstly, rare-earth elements containing 10-30%wt and iron containing 70-90%wt are smelted into iron-based alloys, which are ground into powder after crushed into small particles, and then subjected to nitrogen treatment; the treatment temperature is 250-550°C, and the material chemical formula is $R_2Fe_{17}N_{3-\delta}$. However, the nitrogen content and uniformity of the products obtained by this method need to be improved.

[0009] CN 107557551A discloses a preparation method for a samarium-iron-nitrogen series permanent magnet material, which comprises that metastable state samarium-iron alloy is subjected to severe plastic deformation and then subjected to nitrogen treatment and annealing crystallization treatment, which facilitates entrance and diffusion of nitrogen atoms, and can remarkably improve the nitriding amount and uniformity of the alloy. However, the process of this scheme is too complicated, resulting in the increase of preparation cost.

SUMMARY

[0010] An object of the present application is to provide a method for nitriding a rare-earth magnet, and a nitrided rare-earth magnet. The method provided in the present application solves the problems of insufficient nitrogen content and uneven distribution of nitrogen element in the rare-earth magnet after nitrogen treatment.

[0011] To achieve the object, the present application adopts the technical solutions below.

[0012] In a first aspect, the present application provides a method for nitriding a rare-earth magnet, which includes the following steps:

(1) vacuumizing, adding a nitrogen source into a reactor with a rare-earth magnet, performing a three-stage heating treatment to obtain a semi-finished product; the heating temperature of the three-stage heating treatment is increased stage by stage;

(2) heating the semi-finished product in step (1) to a first set temperature, then adjusting the temperature to a second set temperature and vacuumizing, introducing inert gas, and performing heat preservation at the second set temperature to obtain a nitrided rare-earth magnet; the second set temperature is lower than the first set temperature.

[0013] In the method provided by the present application, the three-stage heating treatment is a main nitriding process, which can ensure that nitrogen-containing gas molecules adsorbed on the surface of the alloy are diffused into the interior the alloy (rare-earth magnet) to form a nitrogen-containing alloy (i.e., semi-finished product), so that nitrogen element can rationally utilize diffusion kinetics after adsorption onto the surface of powder to improve the penetration depth of nitrogen element, thus increase the nitrogen content; heating at the first set temperature is a homogenization treatment step, which can further promote a adsorption of nitrogen element and diffuse the nitrogen into the alloy, so that the distribution of nitrogen element is more uniform in the alloy; and heating at the second set temperature makes nitrogen element diffuse uniformly in the alloy.

[0014] In the method provided by the present application, the method of adjusting the temperature to the second set temperature and vacuumizing in step (2) may be directly cooling to the second set temperature and then vacuumizing, or cooling to room temperature, vacuumizing, and then heating to the second set temperature.

[0015] In the method provided by the present application, three different parts may be selected on the obtained nitrided rare-earth magnet to measure a mass fraction of nitrogen element with a nitrogen content detection device to investigate whether the distribution of nitrogen element is uniform.

[0016] In the present application, the rare-earth magnet in step (1) may be block or powder, and powder is optionally selected, which has better effect.

[0017] The following are optional technical solutions of the present application, but not as limitations of the technical solutions provided in the present application. Through the following technical solutions, the technical purposes and advantages of the present application can be better achieved and realized.

[0018] As an optional technical solution of the present application, a general formula of the rare-earth magnet in step (1) is ReTm, wherein Re is a rare-earth metal and Tm is a 3d transition group element and/or 4d transition group element. The 3d transition group element includes at least one of Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu or Zn; the 4d transition group element includes at least one of Y, Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag or Cd.

[0019] Optionally, a general formula of the nitrided rare-earth magnet is ReTmN. In the present application, a specific ratio of Re and Tm is not limited, as long as it can be phased.

[0020] Optionally, the vacuumizing in step (1) reduces a pressure of the reactor to less than 1000 Pa, such as 900 Pa, 500 Pa, 100 Pa, 50 Pa, 10 Pa or 5 Pa, optionally less than or equal to 10 Pa.

[0021] Optionally, the nitrogen source in step (1) includes a gaseous nitrogen source and/or a solid nitrogen source.

[0022] Optionally, the gaseous nitrogen source is a nitrogen-containing gas.

[0023] Optionally, the gaseous nitrogen source includes nitrogen and/or ammonia.

[0024] Optionally, the gaseous nitrogen source further includes hydrogen.

[0025] Optionally, the solid nitrogen source includes ammonium bicarbonate.

[0026] Optionally, the nitrogen source in step (1) is a gaseous nitrogen source, and a pressure of the gaseous nitrogen source in the reactor is 0.001-10 MPa, such as 0.001 MPa, 1 MPa, 2 MPa, 5 MPa, 8 MPa or 10 MPa, optionally 0.01-2 MPa.

[0027] Optionally, the nitrogen source in step (1) is a solid nitrogen source, before the three-stage heating treatment, a protective gas is introduced into the reactor to a pressure of 0.001-1 MPa, such as 0.001 MPa, 1 MPa, 2 MPa, 5 MPa, 8 MPa or 10 MPa, optionally 0.01-2MPa.

[0028] As an optional technical solution of the present application, a first-stage temperature of the three-stage heating treatment in step (1) is 330-470 K, such as 330 K, 350 K, 380 K, 400 K, 420 K, 450 K or 470 K, optionally 370-420 K.

[0029] Optionally, a first-stage heat preservation time of the three-stage heating treatment in step (1) is 0-24 h but excluding 0, such as 0.1 h, 0.5 h, 1 h, 5 h, 10 h, 20 h or 24 h, optionally 0.1-5 h.

[0030] In the present application, a purpose of a first-stage heating in the three-stage heating in step (1) is to remove an adsorbed gas, especially oxygen, from ReTm alloy to avoid the oxidation of the alloy during subsequent nitriding.

[0031] As an optional technical solution of the present application, a second-stage temperature of the three-stage heating treatment in step (1) is 670-730 K but excluding 730 K, such as 670 K, 680 K, 690 K, 700 K, 710 K or 720 K, optionally 690-710K.

[0032] Optionally, a second-stage heat preservation time of the three-stage heating treatment in step (1) is 0-24 h but excluding 0, such as 0.1 h, 0.5 h, 1 h, 5 h, 10 h, 20 h or 24 h, optionally 0.1-5 h.

[0033] In the present application, an effect of a second-stage heating in the three-stage heating in step (1) is to preheat ReTm and N element atmosphere and adsorb, so that nitrogen element atmosphere is fully adsorbed on the surface of ReTm.

[0034] As an optional technical solution of the present application, a third-stage temperature of the three-stage heating treatment in step (1) is 730-830 K but excluding 830 K, such as 730 K, 740 K, 750 K, 760 K, 770 K, 780 K, 790 K, 800 K, 810 K or 820 K, optionally 750-780 K.

[0035] Optionally, a third-stage heat preservation time of the three-stage heating treatment in step (1) is 0-24 h but excluding 0, such as 0.1 h, 0.5 h, 1 h, 5 h, 10 h, 20 h or 24 h, optionally 0.1-5 h.

[0036] In the present application, a purpose of a third-stage heating in the three-stage heating in step (1) is to adsorb gas molecules on the surface of the alloy, which can enter the inside of the alloy through diffusion to form a ReTmN alloy.

[0037] As an optional technical solution of the present application, the first set temperature in step (2) is 830-860K but excluding 860 K, such as 830 K, 835 K, 840 K, 845 K or 850 K, optionally 830-840K. In the present application, if the first set temperature is too high, it will cause ReTmN to decompose into ReN and Tm-phase, and the product will deviate from the ideal composition; if the first set temperature is too low, the N content will be low and uniform.

[0038] Optionally, in step (2), a heating time to the first set temperature is 0-24 h but excluding 0, such as 1 h, 5 h, 10 h, 15 h, 20 h or 24 h, optionally 0.5-5 h.

[0039] As an optional technical solution of the present application, the vacuumizing in step (2) reduces a pressure of the reactor to less than 1000 Pa, such as 900 Pa, 500 Pa, 100 Pa, 50 Pa, 10 Pa or 5 Pa, optionally less than or equal to 10 Pa.

[0040] Optionally, a pressure of the reactor is adjusted to 0.001-1 MPa by introducing inert gas in step (2), such as 0.001 MPa, 0.01 MPa, 0.5 MPa or 1 MPa.

[0041] Optionally, the second set temperature in step (2) is 600-700 K, such as 600 K, 610 K, 620 K, 630 K, 640 K, 650 K, 660 K, 670 K, 680 K, 690 K or 700 K, optionally 620-670 K.

[0042] Optionally, a time of the heat preservation at the second set temperature in step (2) is 0-24 h but excluding 0, such as 1 h, 5 h, 10 h, 15 h, 20 h or 24 h, optionally 0.5-5 h.

[0043] As an optional technical solution of the present application, step (2) further includes cooling to room temperature after the heat preservation at the second set temperature.

[0044] As a further optional technical solution of the method described in the present application, the method includes the following steps:

> (1) placing a rare-earth magnet in a reactor, vacuumizing to less than or equal to 1000 Pa, introducing a nitrogen-containing gas, heating to 370-420 K and performing heat preservation for 0.1-5 h, heating to 690-710 K and performing heat preservation for 0.1-5 h, and heating to 750-780 K and performing heat preservation for 0.1-5 h to obtain a semi-finished product;
>
> (2) heating the semi-finished product in step (1) to 830-840 K for 0.5-5 h, then adjusting a temperature to 620-670 K and vacuumizing to reduce a pressure of the reactor to less than or equal to 1000 Pa, introducing inert gas to adjust a pressure of the reactor to 0.001-1 MPa, performing heat preservation at the second set temperature for 0.5-5 h, and cooling to room temperature to obtain a nitrided rare-earth magnet.

[0045] In a second aspect, the present application provides a nitrided rare-earth magnet obtained by the nitriding method of the rare-earth magnet according to the first aspect.

[0046] Compared with the prior art, the present application has the following beneficial effects.

[0047] The method of nitriding a rare-earth magnet provided in the present application, which can make the distribution of nitrogen elements in the magnet more uniform when improving the nitrogen content, solves the problems of insufficient nitrogen content and uneven distribution of nitrogen element after nitriding the rare-earth magnet in the prior art.

DETAILED DESCRIPTION

[0048] In order to better illustrate the present application and facilitate the understanding of the technical solutions of the present application, the present application will be described in further detail below. The following embodiments are only simple examples of the present application, and do not represent or limit the protection scope of the present application. The protection scope of the present application is subject to the claims.

[0049] The following are typical but non-limiting examples of the present application.

Example 1

[0050] In this example, nitridation of a rare earth magnet was carried out as follows:

(1) Sm_2Fe_{17} alloy block was put into a nitriding furnace, a pressure was dropped to 95 Pa by vacuumizing, the vacuum system was closed, and 1.2 MPa ammonia was introduced;

(2) a temperature was heated up to 450 K and performed heat preservation for 2 hours;

(3) after the heat preservation at the temperature of 450 K, the temperature was heated up to 710 K and performed heat preservation for 1 hour;

(4) after the heat preservation at the temperature of 710 K, the temperature was heated up to 780 K and performed heat preservation for 5 hours, and a semi-finished product was obtained;

(5) after the heat preservation at the temperature of 780 K, the temperature was heated to 850 K for 1 hour;

(6) after the heat preservation at the temperature of 850 K, the temperature was cooled to 680 K, gas in the nitriding furnace was pumped out, the pressure was dropped to 90 Pa by vacuumizing, then 0.05 MPa argon was introduced, and the temperature was performed heat preservation for 3 hours; and

(7) after the heat preservation at the temperature of 680 K, the temperature was cooled to room temperature, a nitrided SmFeN alloy was taken out, and three different parts of the material were taken to measure the mass fraction of nitrogen with a nitrogen content detection device. The test results are shown in Table 1.

Example 2

[0051] In this example, nitridation of a rare earth magnet was carried out as follows:

(1) Sm_2Fe_{17} alloy powder with particle size of 0.1 μm-10 mm was put into a nitriding furnace, a pressure was dropped to 9 Pa by vacuumizing, the vacuum system was closed, and 1.2 MPa ammonia was introduced;

(2) a temperature was heated up to 420 K and performed heat preservation for 2 hours;

(3) after the heat preservation at the temperature of 420 K, the temperature was heated up to 680 K and performed heat preservation for 1 hour;

(4) after the heat preservation at the temperature of 680 K, the temperature was heated up to 750 K and performed heat preservation for 4 hours;

(5) after the heat preservation at the temperature of 750 K, the temperature was heated up to 830 K for 1 hour, and a semi-finished product was obtained;

(6) after the heat preservation at the temperature of 830 K, the temperature was cooled to 670 K, gas in the nitriding furnace was pumped out, the pressure was dropped to 9 Pa by vacuumizing, then 0.03 MPa argon was introduced, and the temperature was performed heat preservation for 1 hour; and

(7) after the heat preservation at the temperature of 670 K, the temperature was cooled to room temperature, nitrided SmFeN alloy powder was taken out, and three different parts of the material were taken to measure the mass fraction of nitrogen with a nitrogen content detection device. The test results are shown in Table 1.

Example 3

[0052] Except for step (6), the others are the same as those of Example 2. Step (6) of this example was to cool to room temperature after the heat preservation at the temperature of 830 K, pump out the gas in the nitriding furnace, vacuumize until the pressure drops below 10 Pa, then introduce 0.03MPa argon, heat to 670 K, and perform heat preservation for 1 hour.

Example 4

[0053] Except for step (1), the others were the same as those of Example 2. Step (1) of this example was to close the vacuum system and introduce 1.2 MPa mixture gas of ammonia and hydrogen (mixed molar ratio 1:1).

Example 5

[0054] Except for replacing Sm_2Fe_{17} alloy powder with $SmFe_9$ alloy powder, other conditions were the same as those of Example 2 to prepare an SmFeN alloy.

Example 6

[0055] Except for replacing Sm_2Fe_{17} alloy powder with Ce_2Fe_{17} alloy powder, other conditions were the same as those of Example 2 to prepare a CeFeN alloy.

Example 7

[0056] Except for replacing Sm_2Fe_{17} alloy powder with Nd_2Fe_{17} alloy powder, other conditions were the same as those of Example 2 to prepare an NdFeN alloy.

Example 8

[0057] Except for replacing Sm_2Fe_{17} alloy powder with $NdFe_{12}$ alloy powder, other conditions were the same as those of Example 2 to prepare an NdFeN alloy.

Example 9

[0058] In this example, nitridation of a rare earth magnet was carried out as follows:

(1) Sm_2Fe_{17} alloy powder with particle size of 0.1 μm-10 mm was put into a nitriding furnace, a pressure was dropped to 5 Pa by vacuumizing, the vacuum system was closed, and a 0.01 MPa mixture gas of nitrogen and ammonia (mixed molar ratio 1:1) was introduced;

(2) a temperature was heated up to 400 K and performed heat preservation for 0.1 hour;

(3) after the heat preservation at the temperature of 400 K, the temperature was heated up to 700 K and performed heat preservation for 0.1 hour;

(4) after the heat preservation at the temperature of 700 K, the temperature was heated up to 770 K and performed heat preservation for 0.1 hour;

(5) after the heat preservation at the temperature of 770 K, the temperature was heated up to 840 K for 0.5 hour, and a semi-finished product was obtained;

(6) after the heat preservation at the temperature of 840 K, the temperature was cooled to 650 K, gas in the nitriding furnace was pumped out, the pressure was dropped to 5 Pa by vacuumizing, then 0.01 MPa argon was introduced and the temperature was performed heat preservation for 0.5 hour; and

(7) after the heat preservation at the temperature of 650 K, the temperature was cooled to room temperature, nitrided SmFeN alloy powder was taken out, and three different parts of the material were taken to measure the mass fraction of nitrogen with a nitrogen content detection device. The test results are shown in Table 1.

Example 10

[0059] In this example, nitridation of a rare earth magnet was carried out as follows:

(1) Sm_2Fe_{17} alloy powder with particle size of 0.1 μm-10 mm was put into a nitriding furnace, a pressure was dropped to 5 Pa by vacuumizing, the vacuum system was closed, and 2 MPa ammonia was introduced;

(2) a temperature was heated up to 370 K and performed heat preservation for 5 hours;

(3) after the heat preservation at the temperature of 370 K, the temperature was heated up to 690 K and performed heat preservation for 5 hours;

(4) after the heat preservation at the temperature of 690 K, the temperature was heated up to 770 K and performed heat preservation for 5 hours;

(5) after the heat preservation at the temperature of 770 K, the temperature was heated up to 835 K for 5 hours, and a semi-finished product was obtained;

(6) after the heat preservation at the temperature of 835 K, the temperature was cooled to 620 K, gas in the nitriding furnace was pumped out, the pressure was dropped to 5 Pa by vacuumizing, then 1 MPa argon was introduced, and the temperature was performed heat preservation for 5 hours; and

(7) after the heat preservation at the temperature of 620 K, the temperature was cooled to room temperature, nitrided SmFeN alloy powder was taken out, and three different parts of the material were taken to measure the mass fraction of nitrogen with a nitrogen content detection device. The test results are shown in Table 1.

Example 11

[0060] Except for step (1) of mixing Sm_2Fe_{17} alloy powder (powder particle size of 0.1 μm-10 mm) with a quality of ammonium bicarbonate in a nitriding furnace, vacuumizing until the pressure drops to 9 Pa, closing the vacuum system, and introducing 1.2 MPa argon, the others were the same as those of Example 2 to prepare an SmFeN alloy.

Comparative Example 1

[0061] The method of this comparative example differs from that of Example 2 only in that the operations of step (2) and step (3) were not performed, and the operation of step (3) was to heat to 750K and perform heat preservation for 4 hours.

Comparative Example 2

[0062] The method of this comparative example differs from that of Example 2 only in that the operation of step (5) was not performed, while directly cooling from 750 K to room temperature in step (6).

Comparative Example 3

[0063] The method of this comparative example differs from that of Example 2 only in that the operation of step (3) was not performed, while step (4) was changed to heat to 750 K and perform heat preservation for 4 hours after the heat preservation at the temperature of 420 K.

Test Method

[0064] According to each examples and comparative examples, three different parts of the obtained nitrided rare-earth magnet were taken out, and the mass fraction of nitrogen was measured by a nitrogen content detection device. The test results are shown in Table 1.

Table 1

No.	Nitrogen Content / % (Part 1)	Nitrogen Content / % (Part 2)	Nitrogen Content / % (Part 3)
Example 1	3.61	3.68	3.65
Example 2	3.83	3.81	3.85
Example 3	3.73	3.71	3.70
Example 4	3.21	3.18	3.25
Example 5	3.36	3.40	3.43

(continued)

No.	Nitrogen Content / % (Part 1)	Nitrogen Content / % (Part 2)	Nitrogen Content / % (Part 3)
Example 6	3.82	3.85	3.80
Example 7	0.95	0.93	0.97
Example 8	0.92	0.95	0.93
Example 9	3.08	3.02	3.05
Example 10	3.90	3.93	3.97
Example 11	3.15	3.12	3.10
Comparative Example 1	2.78	2.75	2.80
Comparative Example 2	2.58	2.70	2.82
Comparative Example 3	2.91	2.95	2.97

[0065] Based on the above examples and comparative examples, it can be seen from that the nitriding method of rare-earth magnets provided by the examples, which can make the distribution of nitrogen element in the magnet more uniform when improving the nitrogen content, solves the problems of insufficient nitrogen content and uneven distribution of nitrogen element after nitriding rare-earth magnets in the prior art.

[0066] The nitrogen content of the products in Example 7 and Example 8 is relatively low due to the different types of rare-earth elements used compared with Example 1, which are not comparable with that of Example 1.

[0067] For Comparative Example 1, there is only one-stage heating and no multi-stage heating in the preparation of the semi-finished product, resulting in incomplete exhaust, insufficient nitrogen adsorption, very low nitrogen content after nitriding, and poor nitriding effect.

[0068] For Comparative Example 2, there is no homogenization treatment (that is not heat at the first set temperature), resulting in great fluctuation and nonuniformity of the nitrogen content.

[0069] For Comparative Example 3, there is only two-stage heating in the preparation of the semi-finished product, resulting in insufficient nitrogen adsorption and low nitrogen content after nitriding.

[0070] The applicant declares that the present application describes the detailed methods of the present application through the above embodiments, but the present application is not limited to the above detailed methods, that is, it does not mean that the present application must rely on the above detailed methods to implement.

Claims

1. A method for nitriding a rare-earth magnet, comprising the following steps:

   (1) vacuumizing, adding a nitrogen source into a reactor with a rare-earth magnet, performing a three-stage heating treatment to obtain a semi-finished product; the heating temperature of the three-stage heating treatment is increased stage by stage;
   (2) heating the semi-finished product in step (1) to a first set temperature, then adjusting the temperature to a second set temperature and vacuumizing, introducing inert gas, and performing heat preservation at the second set temperature to obtain a nitrided rare-earth magnet; the second set temperature is lower than the first set temperature.

2. The method according to claim 1, wherein the first set temperature in step (2) is 830-860 K but excluding 860 K.

3. The method according to claim 1 or 2, wherein a general formula of the rare-earth magnet in step (1) is ReTm, wherein Re is a rare-earth metal, Tm is a 3d transition group element and/or a 4d transition group element.

4. The method according to claim 3, wherein a general formula of the nitrided rare-earth magnet is ReTmN.

5. The method according to any one of claims 1 to 4, wherein the vacuumizing in step (1) reduces a pressure of the reactor to less than or equal to 1000 Pa, optionally, less than or equal to 10 Pa;

optionally, the nitrogen source in step (1) comprises a gaseous nitrogen source and/or a solid nitrogen source;
optionally, the gaseous nitrogen source is a nitrogen-containing gas;
optionally, the gaseous nitrogen source comprises nitrogen and/or ammonia;
optionally, the gaseous nitrogen source further comprises hydrogen;
optionally, the solid nitrogen source comprises ammonium bicarbonate and/or ammonium chloride;
optionally, the nitrogen source in step (1) is a gaseous nitrogen source, and a pressure of the gaseous nitrogen source in the reactor is 0.001-10 MPa, optionally 0.01-2 MPa;
optionally, the nitrogen source in step (1) is a solid nitrogen source, before the three-stage heating treatment, a protective gas is introduced into the reactor to a pressure of 0.001-1 MPa, optionally 0.01-2 MPa.

6. The method according to any one of claims 1 to 5, wherein a first-stage temperature of the three-stage heating treatment in step (1) is 330-470 K, optionally 370-420 K;

optionally, a first-stage heat preservation time of the three-stage heating treatment in step (1) is 0-24 h but excluding 0, optionally 0.1-5 h;
optionally, a second-stage temperature of the three-stage heating treatment in step (1) is 670-730 K but excluding 730 K, optionally 690-710K;
optionally, a second-stage heat preservation time of the three-stage heating treatment in step (1) is 0-24 h but excluding 0, optionally 0.1-5 h;
optionally, a third-stage temperature of the three-stage heating treatment in step (1) is 730-830 K but excluding 830 K, optionally 750-780 K;
optionally, a third-stage heat preservation time of the three-stage heating treatment in step (1) is 0-24 h but excluding 0, optionally 0.1-5 h.

7. The method according to any one of claims 2 to 6, wherein the first set temperature in step (2) is 830-840 K; optionally, in step (2), a heating time to the first set temperature is 0-24 h but excluding 0, optionally 0.5-5 h.

8. The method according to any one of claims 1 to 7, wherein the vacuumizing in step (2) reduces a pressure of the reactor to less than or equal to 1000 Pa, optionally less than or equal to 10 Pa;

optionally, a pressure of the reactor is adjusted to 0.001-1 MPa by introducing inert gas in step (2);
optionally, the second set temperature in step (2) is 600-700 K, optionally 620-670 K;
optionally, a time of the heat preservation at the second set temperature in step (2) is 0-24 h but excluding 0, optionally 0.5-5 h.

9. The method according to any one of claims 1 to 8, wherein step (2) further comprises cooling to room temperature after the heat preservation at the second set temperature.

10. The method according to any one of claims 1 to 9, comprising the following steps:

(1) placing a rare-earth magnet in a reactor, vacuumizing to less than or equal to 1000 Pa, introducing a nitrogen-containing gas, heating to 370-420 K and performing heat preservation for 0.1-5 h, heating to 690-710 K and performing heat preservation for 0.1-5 h, and heating to 750-780 K and performing heat preservation for 0.1-5 h to obtain a semi-finished product;
(2) heating the semi-finished product in step (1) to 830-840 K for 0.5-5 h, then adjusting a temperature to 620-670 K and vacuumizing to reduce a pressure of the reactor to less than or equal to 1000 Pa, introducing inert gas to adjust a pressure of the reactor to 0.001-1 MPa, performing heat preservation at the second set temperature for 0.5-5 h, and cooling to room temperature to obtain a nitrided rare-earth magnet.

11. A nitrided rare-earth magnet obtained by the method for nitriding a rare-earth magnet according to any one of claims 1 to 10.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2021/088065

A. CLASSIFICATION OF SUBJECT MATTER

H01F 1/053(2006.01)i； H01F 1/059(2006.01)i； B22F 1/00(2006.01)i； B22F 9/04(2006.01)i； C23C 8/26(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

H01F; B22F; C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, EPODOC, CNPAT, CNKI, IEEE: 稀土, 钐, 钕, 铈, 铁, 钴, 镍, 过渡, 氮化, 渗氮, 吸氮, 氨, 碳酸氢铵, 碳酸铵, 扩散, 均匀, 匀化, 均一, 升温, 加热, 分段, 多段, 阶梯, rare earth, samarium, Sm, neodymium, Nd, cerium, Ce, iron, Fe, cobal, Co, nickel, Ni, transition, nitriding, absorbing nitrogen, ammonia, NH4HCO3, (NH4)2CO3, ammonium bicarbonate, ammonium carbonate, diffusion, uniformity, heat, segment

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CN 104384493 A (ZHEJIANG UNIVERSITY OF TECHNOLOGY) 04 March 2015 (2015-03-04) description, paragraphs [0002]-[0061], and figure 1	1-11
A	CN 107507687 A (GRIREM ADVANCED MATERIALS CO., LTD.) 22 December 2017 (2017-12-22) entire document	1-11
A	CN 106960712 A (NORTH CHINA UNIVERSITY OF SCIENCE AND TECHNOLOGY) 18 July 2017 (2017-07-18) entire document	1-11
A	CN 102468028 A (DAIDO STEEL CO., LTD.) 23 May 2012 (2012-05-23) entire document	1-11
A	EP 1993110 A1 (SEIKO INSTRUMENTS INC.) 19 November 2008 (2008-11-19) entire document	1-11
A	US 6383406 B1 (CHEMAT TECHNOLOGY, INC.) 07 May 2002 (2002-05-07) entire document	1-11

☐ Further documents are listed in the continuation of Box C. ☑ See patent family annex.

* Special categories of cited documents:	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search **18 June 2021**	Date of mailing of the international search report **07 July 2021**
Name and mailing address of the ISA/CN **China National Intellectual Property Administration (ISA/CN)** **No. 6, Xitucheng Road, Jimenqiao, Haidian District, Beijing 100088, China** Facsimile No. **(86-10)62019451**	Authorized officer Telephone No.

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2021/088065

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
CN 104384493 A	04 March 2015	CN 104384493 B	17 August 2016
CN 107507687 A	22 December 2017	CN 107507687 B	27 December 2019
CN 106960712 A	18 July 2017	CN 106960712 B	02 October 2018
CN 102468028 A	23 May 2012	CN 102468028 B	20 April 2016
		JP 5668426 B2	12 February 2015
		JP 2012106264 A	07 June 2012
EP 1993110 A1	19 November 2008	CN 101306467 A	19 November 2008
		JP 2008283141 A	20 November 2008
		US 2012164019 A1	28 June 2012
US 6383406 B1	07 May 2002	None	

Form PCT/ISA/210 (patent family annex) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- CN 101699578 A **[0008]**
- CN 107557551 A **[0009]**