(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 1 993 110 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
19.11.2008 Bulletin 2008/47

(51) Int Cl.:
H01F 1/059 *(2006.01)* *H01F 41/02* *(2006.01)*

(21) Application number: **08251659.2**

(22) Date of filing: **09.05.2008**

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MT NL NO PL PT RO SE SI SK TR
Designated Extension States:
AL BA MK RS

(30) Priority: **14.05.2007 JP 2007128488**

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(54) **Method of manufacturing rare-earth magnet powder and method of manufacturing rare-earth bonded magnet**

(57) A method of manufacturing rare-earth magnet powder having excellent magnetic properties, and a method of manufacturing a rare-earth bond magnet are provided. A nitriding step is performed, in which when nitrided rare-earth magnet powder is produced, rare-earth-element/transition metal-based alloy powder is irradiated with a microwave at an atmosphere containing nitrogen atoms, so that the nitrogen atoms are allowed to enter into a crystal lattice.

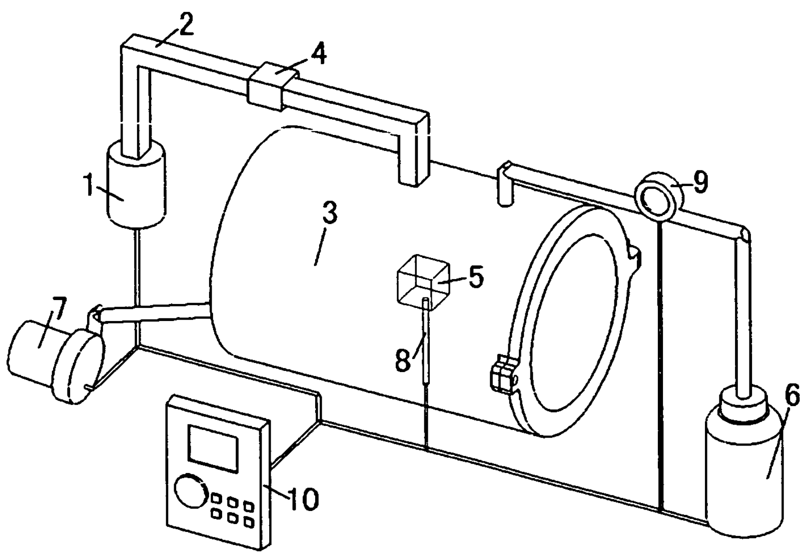

Fig. 1

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Description

[0001] The present invention relates to a method of manufacturing rare-earth magnet powder, and a method of manufacturing a rare-earth bond magnet.

[0002] Recently, much attention has been paid to a rare-earth magnet as a magnet having excellent magnetic properties. Among various magnet materials for the rare-earth magnet, a magnet produced by nitriding SmFe alloy or the like (hereinafter, called nitride compound magnet) is particularly focused with attention as a magnet material having high Curie point and excellent magnetic anisotropy.

[0003] Method of manufacturing a nitride compound magnet having a rare earth element as a main component typically has steps ofalloymelting, crushing, nitriding, press forming, sintering, machining, magnetization and the like. Typically, innitriding, alloy powder is subjected to heating under a gas atmosphere of N_2, NH_3 or the like, or a mixed gas atmosphere of such gas and H_2 gas, and thereby nitrogen is introduced in a crystal lattice through a solid-gas reaction. In such nitriding, while the alloy powder is easily nitrided at high temperature, when the powder is subjected to heating at high temperature of about 650°C, decomposition of a nitride compound occurs, resulting has formation of a soft magnetic, α-Fc phase. Therefore, it has been necessary that the nitriding is performed for a long time at low temperature.

[0004] To cope with the problem, JP-A-5-109518 describes a method, in which alloy powder is heated at a condition of high pressure of 1.5 atm to 300 atm and a temperature of 500°C or less, so that decomposition of a nitride compound is suppressed. JP-A-5-135978 describes a method, in which nitriding is performed by plasma sintering at high pressure (2 atm or more), so that decomposition of a nitride compound is suppressed, and processing time is reduced while it has been long so far.

[0005] JP-A-11-87118 describes a method, in which when rare-earth magnet powder having fine grain diameter is nitrided under an atmosphere of a mixed gas including NH_3 and H_2, partial pressure of each gas is controlled so that excessive nitriding is suppressed. Furthermore, Imaoka et al. "Magnetic Properties and Nitriding Process of Sm2Fe17Nx" Transaction A of The Institute of Electrical Engineers of JAPAN, vol. 113, No. 4, pp 276-285, September 28, 1992 describes a method in which nitriding is performed by an ammonia-hydrogen mixing method to improve nitriding efficiency.

[0006] However, in a method of performing heating at high pressure as the method described in JP-A-5-10 9518 or JP-A-5-135978, there is a problem that nitriding is inadequate, so that a region being not nitrided is formed in a rare-earth-element/transition metal-based magnet material, consequently the magnetic material is nonuniformly nitrided.

[0007] Moreover, in the method of controlling partial pressure of the mixed gas during nitriding as described in JP-A-11-87118, crystal grain diameter of alloy particles as a nitriding object is small, 50 nm or less, and therefore even if partial pressure of each of NH_3 and H_2 is controlled, the alloy particles may be nonuniformly nitrided.

[0008] Furthermore, in the method using the ammonia-hydrogen mixing method described in the "Magnetic Properties and Nitriding Process of Sm2Fe17Nx" Transaction A of The Institute of Electrical Engineers of JAPAN, vol. 113, No. 4, although processing time can be reduced, since heating is performed while ammonia and hydrogen are made to flow at a predetermined flow rate respectively, a region being nonuniform in the amount of ammonia or hydrogen is sometimes formed in a tubular electric furnace. As a result, nitriding easily proceeds only in a particular portion of the rare-earth-element/transition metal-based magnet material, resulting in nonuniform nitriding. Moreover, when hydrogen remains in a magnet, the magnet becomes gradually significantly brittle in a long-term use condition, which is not preferable.

[0009] As equipment for performing each kind of nitriding as above, a large external furnace is typically used. In such a case, alloy powder is easily oxidized, and thus a soft magnetic phase is easily formed. Therefore, a measure is taken, for example, high-purity nitrogen is filled into the furnace, or nitrogen gas in the furnace is repeatedly substituted or repeatedly evacuated, so that oxidation is suppressed. However, in this case, processing time is increased and thus production efficiency is reduced, inaddition, cost is extremely increased. Moreover, even if the nitrogen gas is substituted or evacuated, oxygen is hard to be perfectly removed from the inside of the alloy powder.

[0010] In this way, each of the methods has various problems, and each value of magnetic properties of the rare-earth-element/transition metal-based magnet nitrided by eachmethod is still considerably low compared with a theoretical value at present.

[0011] The invention was made in the light of the above problems, and an object of the invention is to provide a method of manufacturing rare-earth magnet powder having excellent magnetic properties, and a method of manufacturing a rare-earth bond magnet.

[0012] Another object of the invention is to provide a method of manufacturing rare-earth magnet powder, and a method of manufacturing a rare-earth bond magnet, by each of which processing time in a nitriding step can be reduced.

[0013] To solve the problem, a first aspect of the invention is summarized by having a nitriding step in which rare-earth-element/transition metal-based alloy powder is irradiated with a microwave in an atmosphere containing nitrogen atoms, so that the nitrogen atoms are allowed to enter into a crystal lattice.

[0014] A second aspect of the invention is summarized in that in the method of manufacturing rare-earth magnet powder of the first aspect, a homogenization step is further performed after the nitriding step, in which the rare-earth magnet powder is heated in an inert gas.

[0015] A third aspect of the invention is summarized in that in the method of manufacturing rare-earth magnet powder of an above aspect, the microwave irradiated to the alloy powder has a frequency of 1 GHz to 30 GHz.

[0016] A fourth aspect of the invention is summarized in that in the method of manufacturing rare-earth magnet powder of an above aspect, the microwave irradiated to the alloy powder has a frequency of 1 GHz to 30 GHz.

[0017] A fifth aspect of the invention is summarized in that in the method of manufacturing rare-earth magnet powder of any one of the first to fourth aspects, average grain diameter of the alloy powder is 2 to 90 μm.

[0018] A sixth aspect of the invention is summarized in that in the method of manufacturing rare-earth magnet powder of any one of the first to fifth aspects, pressure of an atmospheric gas containing nitrogen is 0.1 to 5 MPa in the nitriding step.

[0019] A seventh aspect of the invention is summarized in that in the method of manufacturing rare-earth magnet powder of any one of the first to sixth aspects, microwave irradiation is performed while the rare-earth-element/transition metal-based alloy powder is heated in a temperature range of 250°C to 600°C in the nitriding step.

[0020] An eighth aspect of the invention is summarized in that the rare-earth magnet powder in any one of the first to seventh aspects is mixed with a resinbinder or a metal binder for molding.

[0021] According to the first aspect of the invention, the rare-earth-element/transition metal-based alloy powder is irradiated with the microwave, thereby self -heating of the alloy powder itself, rapid heating, and selective heating can be performed. Therefore, processing time taken for nitriding can be reduced. Moreover, the powder can be uniformly nitrided even at the inside thereof, so that rare-earth magnet powder having high magnetic properties can be obtained.

[0022] According to the second aspect of the invention, homogenization, in which the rare-earth magnet powder subjected to nitriding is heated, is performed, thereby nitrogen atoms can be moved to stable, interstitial sites in a crystal lattice.

[0023] According to the third and fourth aspects of the invention, since the microwave irradiated in the nitriding step is in the frequency of 1 GHz to 30 GHz, a phenomenon that solid-phase diffusion preferentially proceeds rather than nitriding can be suppressed, and the powder can be uniformly nitrided even at the inside thereof.

[0024] According to the fifth aspect of the invention, since grain diameter of the alloy powder as a nitriding object is 2 to 90 μm, oxidation and excessive nitriding of the alloy powder can be suppressed, and the alloy powder can be uniformly nitrided.

[0025] According to the sixth aspect of the invention, since atmosphere in the nitriding step is in a pressure of is 0.1 to 5 MPa, the alloy powder can be uniformly nitrided, and formation of an amorphous state in alloy due to excessive nitriding caused by excessive pressure can be prevented.

[0026] According to the seventh aspect of the invention, since microwave irradiation is performed while the alloy powder is heated in the nitriding step, nitriding of the alloy powder can be more efficiently accelerated.

[0027] According to the eighth aspect of the invention, since a rare-earth bond magnet is produced by using the rare-earth magnet powder subjected to nitriding, and a resin binder or a metal binder, a bond magnet having excellent magnetic properties can be obtained.

[0028] Embodiments of the present invention will now he described by way of further example only and with reference to the accompanying drawing., in which:

Fig. 1 shows a schematic diagram showing an embodiment of the invention.

[0029] A microwave oscillating tube 1 that oscillates a microwave is connected to an applicator 3 via a waveguide 2. The microwave oscillated by the microwave oscillating tube 1 is transmitted to the applicator 3 through the waveguide 2. The waveguide 2 has an isolator 4. The isolator 4 transmits a microwave on the waveguide 2 only to a direction of the applicator 3, and absorbs a microwave transmitted in an opposite direction.

[0030] A specimen 5 is placed in the applicator 3 and irradiated with the microwave. The applicator 3 is a closed vessel made of metal, and is formed so as to prevent leakage of the microwave to the outside. Moreover, the applicator 3 is connected with a gas supply source 6 for introducing an inert gas such as nitrogen. Furthermore, the applicator 3 is connected with a pump 7 for exhausting atmosphere from the inside thereof.

[0031] The specimen 5 is connected to a thermocouple 8, so that temperature change of the specimen 5 associated with microwave irradiation can be measured. The applicator 3 has a pressure gauge 9, so that internal pressure can be measured. The microwave oscillating tube 1, gas supply source 6, pump 7, thermocouple 8, and pressure gauge 9 are connected to a controller 10, and thus controlled respectively. Thus, atmosphere and pressure within the applicator, increase in temperature of the specimen, and the like can be controlled by the controller 10.

[0032] As the microwave oscillating tube 1, magnetron, gyrotron, klystron and the like can be used.

[0033] A method of manufacturing the rare-earth-element/transition metal-based (hereinafter, called R-TM-based) magnet powder of the invention, and a method of manufacturing a rare-earth bond magnet using the powder are described for each step below. R is configured by at least one or two elements among rare earth elements, and TM is configured by at least one or two elements among transition elements.

(1) Manufacturing of R-TM-based alloy powder

[0034] As the rare earth element configuring the R-TM-based alloy of the invention, lanthanide elements (La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu) including Y (yttrium) can be used. Particularly, when Pr, Nd or Sm is used, magnetic properties can be remarkably improved. Moreover, when at least two kinds of rare earth elements are used in combination with each other, residual magnetic flux density and coercive force of the magnetic properties can be improved.

[0035] Moreover, the rare earth element preferably occupies 5 to 30 at% of all elements. When it is less than 5 at%, a soft magnetic phase is increased, which is not preferable. When it is more than 30 at%, since the content of transition metal is decreased, an adequate amount of main phase is not formed. In addition, since a ratio of a nonmagnetic phase having a considerably high content of rare earth element is increased, the magnetic properties are significantly degraded, and consequently magnetic properties cannot be adequately obtained.

[0036] As the transitionmetal used for the R-TMalloy, any element can be used, but Fe is particularly preferable, and furthermore Mn, Co and Ni may be mixed for thermal stability. Even in this case, alloy can be obtained without causing degradation in the magnetic properties. Moreover, the transition metal desirably occupies 30 to 80 at% of all elements. When it is less than 30 at%, saturation magnetization is reduced. When it is more than 80 at%, high coercive force is not obtained.

[0037] As the methodof manufacturing the R-TM-based alloypowder, while it is not particularly limited, for example, a melt-casting method or a liquid quenching method can be used. In the case of a typical melt-casting method, the rare-earth metal, transition metal, and additive metal are mixed in a predetermined mixing ratio and then subjected to high-frequency melting in an inert-gas atmosphere, and an obtained alloy ingot is subjected to heat treatment, and then crushed into a predetermined grain diameter by a crusher such as a jaw crusher, a jet mill, or an attritor.

[0038] In the liquid quenching method, in contrast to the alloy ingot produced as above, molten alloy is discharged onto a roll being rotated at high speed so as to be contacted to an outer circumferential surface of the roll, thereby the molten alloy is quenched to produce an alloy ribbon. The alloy ribbon is crushed into a predetermined grain diameter by the crusher. Even if C, B or the like is mixed in the alloy as an inevitable impurity during such melting, no particular problem occurs.

[0039] In this case, the grain diameter is preferably 2 to 90 μm in average. When the average diameter is less than 2 μm, alloy particles are easily oxidized and may be excessively nitrided in a subsequent nitriding step. Moreover, increase in density due to agglomeration or spring back is not achieved during producing a bond magnet, resulting in degradation in magnetic properties. Moreover, when the average diameter is more than 90 μm, a particle is not nitrided at the inside thereof , leading to nonuniform nitriding, which induces degradation in magnetic properties.

(2) Nitriding

[0040] When R-TM-based alloy powder is obtained by the above method, the R-TM-based alloy powder is subjected to nitriding. In the nitriding, the R-TM-based alloy powder is subjected to irradiation of a microwave at an atmosphere containing nitrogen atoms. In this case, if the microwave irradiation is performed at normal pressure, for example, when grain diameter of the alloy powder is relatively large, a nitrided region and a non-nitrided region may be unevenly formed in the alloy powder, which causes variation in magnetic properties. Therefore, a pressurized atmosphere is preferred to eliminate the non-nitrided region, thereby even in alloy powder having a large grain diameter, uniform nitriding can be achieved due to a synergetic effect of gas pressure and microwave irradiation.

[0041] As the pressurizing gas, air, nitrogen, or a mixed gas such as air and nitrogen, nitrogen and an inert gas (excluding nitrogen), and air and an inert gas can be used. The pressure given by the gas is preferably 0.1 to 5 Mpa depending on an irradiation condition of the microwave. When the pressure is less than 0.1 Mpa, nitrogen atoms may not enter into the inside of an alloy particle, and remained only in a surface. When it is more than 5 Mpa, formation of an amorphous state may be induced in the alloy particle due to excessive nitriding. Furthermore, in the nitriding, hydrogen can be prevented from remaining in the alloy powder by avoiding use of the gas of ammonia, hydrogen and the like.

[0042] In the nitriding, the R-TM-based alloy powder is preferably irradiated with a microwave while it is heated to a temperature in a range of 250 to 600°C, thereby the alloy powder can be optimallynitrided. When heating temperature is less than 250°C, proceeding speed of nitriding is reduced. When heating temperature is more than 600°C, alloy may be decomposed into a nitride of a rare-earth element and a soft magnetic phase, which is not preferable.

[0043] Frequency of the microwave irradiated to the alloy powder is preferably 1 GHz to 30 GHz. When the frequency is less than 1 GHz, a discharge phenomenon tends to occur, so that solid-phase diffusion preferentially proceeds rather than nitriding. When it is more than 30 GHz, since a wavelength is short, penetration depth of the microwave to the inside of a powder particle becomes small, which induces a phenomenon that the particle is not nitrided at the inside thereof. Output power of the microwave is preferably 5 kW or less to obtain a stable electric field. When it is more 5 kW, expensive equipment is necessary, leading to excessively high cost.

[0044] Nitriding is performed at the above optimized condition, thereby while several hours or more has been required for nitriding using an external furnace in the past, alloy particles can be nitrided within one hour in the nitriding using microwave irradiation.

[0045] Moreover, the rare-earth magnet powder is selectively and rapidly self-heated by microwave irradiation, whereby a magnetic material can be obtained, in which nitrogen is allowed to enter into a metallic lattice while keeping a particle shape.

[0046] Furthermore, since a nitriding reaction selectively proceeds under microwave irradiation, oxidation is not found even in an environment having a comparatively high content of oxygen. Therefore, above effects are particularly exhibited in fine powder that is large in specific surface area, and easily affected by oxidation.

[0047] As a mechanism of a nitriding process using a microwave, stabilization of a reaction product, and activity of a reactant are analytically given, but many points still have not been elucidated, and are under investigation as future issues.

(3) Homogenization

[0048] After the nitriding is finished, homogenization is performed. That is, after the microwave has been irradiated, while the adequate amount of nitrogen has been entered in the surface and the inside of the alloy-powder, insertion sites of nitrogen are still in an unstable state. Therefore, homogenization is performed so that nitrogen is moved to stable sites and stabilized.

[0049] In the homogenization, when the nitrided R-TM-based alloy powder is subjected to heat treatment at 200 to 600°C for 0.5 to 5.0 hours in an inert gas such as nitrogen, coercive force of the magnetic properties can be further improved. Here, if a heat treatment furnace is evacuated to a vacuum, nitrogen is escaped, therefore it is important that heat treatment is performed in the inert gas so as to be not affected by external atmosphere, so that nitrogen atoms are moved to stable sites in a crystal lattice.

[0050] While the homogenization may be performed by the same microwave apparatus as in the nitriding, it may be performed by an external furnace. As hereinbefore, the R-TM-based alloy powder is subj ected to the nitriding using microwave irradiation and the homogenization, thereby nitrogen-interstitial rare-earth magnet powder can be obtained, which has excellent magnetic properties and is stabilized.

(4) Rare-earth bond magnet

[0051] When a bond magnet of the invention is produced, the rare-earth magnet powder obtained in the above way is mixed with a resin binder and thus formed into a compound. In this case, the resin binder to be used is not particularly limited, but when thermoplastic resin is used for the binder, resin having good moldability can be used, for example, engineering plastic resin, including polyamide resin, polyacetal resin, polycarbonate resin, polyphenylene resin, aromatic polyester or elastomer. When high heat resistance is required, PPS, PEEK, LCP, fluorine resin or the like can be used. The thermoplastic resin and the rare-earth magnet powder are melted and mixed, and then pelletized. Thus, the pelletized resin and powder are subjected to injection molding or extrusion molding.

[0052] When thermosetting resin is used for the resin binder, unsaturated polyester resin, phenol resin, melamine resin or the like can be used. In this case, an organic solvent or the like is preferably used for mixing and deaeration before preparing the pelletizedpowder, so that the thermosetting resin and an additive are particularly uniformly mixed.

[0053] In the case that the thermosetting resin is used, when a compact obtained by compression molding is subjected to microwave irradiation, the rare-earth magnet powder is rapidly and selectively self-heated, and thereby the compact can be heated to a target temperature in a few minutes. Heat generated by the magnet powder is transferred to the thermosetting resin around the magnet, consequently the resin can be instantaneously cured.

[0054] The magnet powder may be mixed with a metal binder instead of the resin binder, then the mixed powder may be subjected to compression molding, thereby a rare-earth bond magnet having excellent heat resistance can be obtained. In this case, low-melting-point metal or low-melting-point alloy, which is selectively heated by a microwave, can be used for the metal binder, and for example, metal such as Mg, Al, Cu, Zn, Ga, Pb, Sn, Bi and the like, or alloy using the respective kinds of metal is preferably used. Moreover, the rare-earth magnet powder is covered approximately uniformly by the metal binder by using a vacuum deposition, chemical vapor deposition, physical vapor deposition, electroplating, a fusion method or the like, thereby a high-density compression molding product can be obtained. When a bond magnet is produced in this way, coarsening of crystal grains is suppressed, consequently resin bonding can be achieved between magnet particles without oxidizing the particles.

[0055] In the case of rare-earth magnet powder prepared by the melt casting method, since the powder has magnetic anisotropy, a magnetic circuit is incorporated in a die of a molding machine in a molding step, and an orientation magnetic field is applied to the powder so as to align crystal orientations. Thus, magnetic properties of the powder can be maximally exhibited, consequently a rare-earth bond magnet having high magnetic flux density can be manufactured.

[0056] In the case of rare-earth magnet powder prepared by the liquid quenching method, since molding is performed without applying an orientation magnetic field, a molding cycle is reduced, consequently a rare-earth bond magnet, which is high in productivity and inexpensive, can be manufactured.

[0057] According to the embodiment, the following effects can be obtained.

(1) In the embodiment, to produce the nitrided rare-earth magnet, the R-TM-based alloy powder is irradiated with a microwave at an atmosphere contain-

ing nitrogen atoms so that the nitrogen atoms are allowed to enter into a crystal lattice. Therefore, since the alloy powder itself can be selectively and rapidly self-heated, processing time taken for nitriding can be reduced, and furthermore not only a surface of the powder but also the inside thereof can be uniformly nitrided, and consequently rare-earth magnet powder having high magnetic properties can be obtained.

(2) In the embodiment, a homogenization step is further performed, in which a nitrided rare-earth magnet is heated in an inert gas. Therefore, nitrogen atoms in an unstable state can be moved to stable, interstitial sites in a crystal lattice.

(3) In the embodiment, the microwave irradiated to the alloy powder in the nitriding step is made to have a frequency of 1 GHz or more and 30 GHz or less. Therefore, a discharge phenomenon that tends to occur at low frequency can be prevented, and a phenomenon that solid-phase diffusion preferentially proceeds rather than nitriding can be suppressed. Moreover, a wavelength of the microwave is not excessively reduced and made to be an appropriate wavelength, and thereby the microwave can be transmitted to the inside of the alloy powder, so that the inside can be also uniformly nitrided.

(4) In the embodiment, alloy powder having average grain diameter of 2 to 90 μm is nitrided. Therefore, oxidation and excessive nitriding of the alloy powder can be suppressed, and the alloy powder can be uniformly nitrided.

(4) In the embodiment, since pressure of pressurized gas used in the nitriding step is made to be 0.1 to 5 MPa, the alloy powder can be uniformly nitrided due to a synergetic effect of pressurization by the gas and the microwave irradiation, and excessive nitriding due to excessive pressure can be prevented.

(6) In the embodiment, since the rare-earth magnet powder being nitrided by the microwave irradiation is mixed with a resin binder or a metal binder for molding, a bond magnet having excellent magnetic properties can be obtained.

[0058] The aforegoing description has been given by way of example only and it will be appreciated by a person skilled in the art that modifications can be made without departing from the scope of the present invention.

Claims

1. A method of manufacturing rare-earth magnet powder, comprising:

providing rare-earth-element/transition metal-based alloy powder, and
nitriding the powder by irradiating a microwave in an atmosphere containing nitrogen atoms, whereby the nitrogen atoms are enter into a crystal lattice.

2. The method of manufacturing rare-earth magnet powder according to claim 1:

further comprising homogenizing after the nitriding step wherein the rare-earth magnet powder is heated in an inert gas.

3. The method of manufacturing rare-earth magnet powder according to claim or claim 2:

wherein the frequency of the microwave is 1 GHz to 30 GHz.

4. The method of manufacturing rare-earth magnet powder according to any one of the preceding claims:

wherein the average grain diameter of the powder is 2 μm to 90 μm.

5. The method of manufacturing rare-earth magnet powder according to any one of the preceding claims:

wherein the pressure of an atmospheric gas containing nitrogen is 0.1 MPa to 5 MPa.

6. The method of manufacturing rare-earth magnet powder according to any one of the preceding claims:

wherein the alloy powder is heated to a temperature in range of 250°C to 600°C in the nitriding step.

7. A method of manufacturing a rare-earth bond magnet:

wherein the rare-earth magnet powder according to any one of the preceding claims, is mixed with a resin binder or a metal binder for molding.

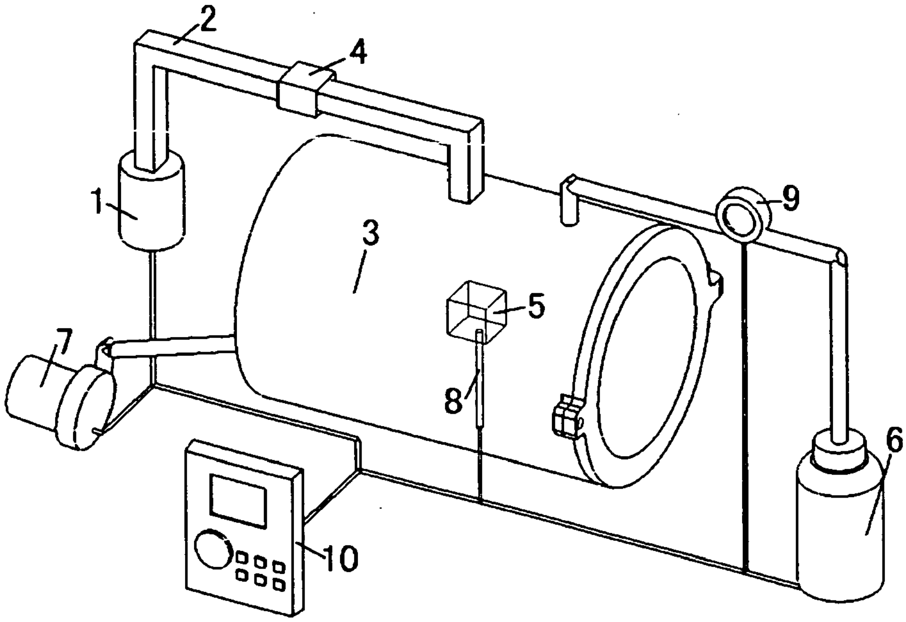

Fig. 1

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EUROPEAN SEARCH REPORT

Application Number
EP 08 25 1659

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
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CATEGORY OF CITED DOCUMENTS

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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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