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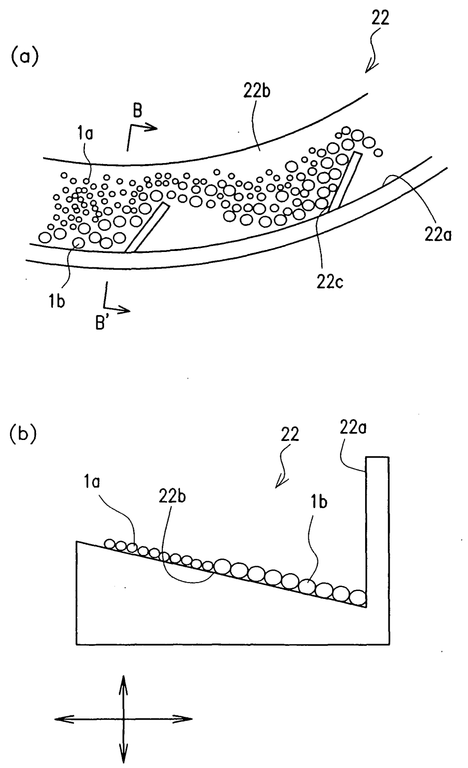
(54) PROCESS AND SYSTEM FOR PRODUCING GRANULATION POWDER OF RARE EARTH ALLOY AND PROCESS FOR PRODUCING SINTERED OBJECT OF RARE EARTH ALLOY

(57) A method for making a granulated powder according to the present invention includes the steps of: preparing a rare-earth alloy powder with remanent magnetization; feeding the powder onto a track 22, which is defined by a side surface 22a and a lower surface 22b that is sloped so as to decrease its height toward the side surface; and setting up vibrations on the track to give the powder kinetic energy, thereby transporting the powder along the length of the track and granulating the powder under a substantially zero magnetic field by utilizing an agglomeration force produced by the remanent magnetization of the powder and a tumbling action produced by the kinetic energy. As a result, a rare-earth alloy granulated powder, which has good flowability and good compactibility and which makes it possible to produce a magnet with excellent magnetic properties, can be obtained.

FIG.2



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Description

TECHNICAL FIELD

[0001] The present invention relates to a method and apparatus for making a rare-earth alloy granulated powder and a method for making a rare-earth alloy sintered body.

BACKGROUND ART

[0002] A rare-earth alloy sintered magnet (permanent magnet) is normally produced by compacting a powder of a rare-earth alloy, sintering the resultant powder compact and then subjecting the sintered body to an aging treatment. Permanent magnets currently used extensively in various applications include rare-earth-cobalt based magnets and rare-earth-iron-boron based magnets. Among other things, the rare-earth-iron-boron based magnets (which will be referred to herein as "R-Fe-B based magnets", where R is one of the rare-earth elements including Y, Fe is iron, and B is boron) are used more and more often in various electronic appliances. This is because an R-Fe-B based magnet exhibits a maximum energy product, which is higher than any of various other types of magnets, and yet is relatively inexpensive.

[0003] An R-Fe-B based sintered magnet includes a main phase consisting essentially of a tetragonal $R_2Fe_{14}B$ compound, an R-rich phase including Nd, for example, and a B-rich phase. In the R-Fe-B based sintered magnet, a portion of Fe may be replaced with a transition metal such as Co or Ni and a portion of boron (B) may be replaced with carbon (C). An R-Fe-B based sintered magnet, to which the present invention is applicable effectively, is described in United States Patents Nos. 4,770,723 and 4,792,368, for example, the entire contents of which are hereby incorporated by reference.

[0004] In the prior art, an R-Fe-B based alloy has been prepared as a material for such a magnet by an ingot casting process. In an ingot casting process, normally, rare-earth metal, electrolytic iron and ferroboron alloy as respective start materials are melted by an induction heating process, and then the melt obtained in this manner is cooled relatively slowly in a casting mold, thereby preparing an alloy ingot.

[0005] Recently, a rapid cooling process such as a strip casting process or a centrifugal casting process has attracted much attention in the art. In a rapid cooling process, a molten alloy is brought into contact with, and relatively rapidly cooled by, a single chill roller, a twin chill roller, a rotating disk or the inner surface of a rotating cylindrical casting mold, thereby making a solidified alloy, which is thinner than an alloy ingot, from the molten alloy. The solidified alloy prepared in this manner will be referred to herein as an "alloy flake". The alloy flake produced by such a rapid cooling process usually has a thickness of about 0.03 mm to about 10 mm. According to the rapid cooling process, the molten alloy starts to be solidified from its surface that has been in contact with the surface of the chill roller. That surface of the molten alloy will be referred to herein as a "roller contact surface". Thus, in the rapid cooling process, columnar crystals grow in the thickness direction from the roller contact surface. As a result, the rapidly solidified alloy, made by a strip casting process or any other rapid cooling process, has a structure including an $R_2Fe_{14}B$ crystalline phase and an R-rich phase. The $R_2Fe_{14}B$ crystalline phase usually has a minor-axis size of about 0.1 μm to about 100 μm and a major-axis size of about 5 μm to about 500 μm. On the other hand, the R-rich phase, which is a non-magnetic phase including a rare-earth element R at a relatively high concentration and having a thickness (corresponding to the width of the grain boundary) of about 10 μm or less, is dispersed on the grain boundary between the $R_2Fe_{14}B$ crystalline phases.

[0006] Compared to an alloy made by the conventional ingot casting process or die casting process (such an alloy will be referred to herein as an "ingot alloy"), the rapidly solidified alloy has been quenched in a shorter time (i.e., at a cooling rate of 10^2 °C /s to 10^4 °C /s). Accordingly, the rapidly solidified alloy has a finer texture and a smaller crystal grain size. In addition, in the rapidly solidified alloy, the grain boundary thereof has a greater area and the R-rich phase is dispersed broadly and thinly over the grain boundary. Thus, the rapidly solidified alloy also excels in the dispersiveness of the R-rich phase. Because the rapidly solidified alloy has these advantageous features, a magnet with excellent magnetic properties can be made from the rapidly solidified alloy.

[0007] An alternative alloy preparation method called "Ca reduction process (or reduction/diffusion process)" is also known in the art. This process includes the processing and manufacturing steps of: adding metal calcium (Ca) and calcium chloride (CaCl) to either the mixture of at least one rare-earth oxide, iron powder, pure boron powder and at least one of ferroboron powder and boron oxide at a predetermined ratio or a mixture including an alloy powder or mixed oxide of these constituent elements at a predetermined ratio; subjecting the resultant mixture to a reduction/diffusion treatment within an inert atmosphere; diluting the reactant obtained to make a slurry; and then treating the slurry with water. In this manner, a solid of an R-Fe-B based alloy can be obtained.

[0008] It should be noted that any small block of a solid alloy will be referred to herein as an "alloy block". The "alloy block" may be any of various forms of solid alloys that include not only solidified alloys obtained by cooling a melt of a material alloy (e.g., an alloy ingot prepared by the conventional ingot casting process or an alloy flake prepared by a rapid cooling process such as a strip casting process) but also a solid alloy obtained by the Ca reduction process.

[0009] An alloy powder to be compacted is obtained by performing the processing steps of: coarsely pulverizing an alloy block in any of these forms by a hydrogen absorption process, for example, and/or any of various mechanical milling processes (e.g., using a disk mill); and finely pulverizing the resultant coarse powder (with a mean particle size of 10 μm to 500 μm) by a dry milling process using a jet mill, for example.

[0010] The R-Fe-B based alloy powder to be compacted preferably has a mean particle size of 1.5 μm to about 6 μm to achieve sufficient magnetic properties. It should be noted that the "mean particle size" of a powder refers to herein an FSSS particle size unless stated otherwise. However, when a powder with such a small mean particle size is used, the resultant flowability, compactibility (including cavity fill density and compressibility) and productivity will be bad.

[0011] To overcome this problem, a method for coating the surface of alloy powder particles with a lubricant was proposed. For example, Japanese Patent Application Laid-Open Publication No. 08-111308 and United States Patent No. 5,666,635 disclose the technique of making an R-Fe-B based alloy fine powder (with a mean particle size of 1.5 μm to 5 μm) by adding 0.02 mass% to 5.0 mass% of a lubricant (including at least one liquefied fatty acid ester) to an R-Fe-B based alloy coarse powder with a mean particle size of 10 μm to 500 μm and then pulverizing the mixture by a jet mill within an inert gas.

[0012] The lubricant not only improves the flowability and compactibility (or compressibility) of the powder but also functions as a binder for increasing the hardness (or strength) of the compact. Nevertheless, the lubricant may also remain as residual carbon in the sintered body to possibly deteriorate the magnetic properties. Accordingly, the lubricant needs to exhibit good binder removability. For example, Japanese Patent Application Laid-Open Publication No. 2000-306753 discloses, as preferred lubricants with good binder removability, depolymerized polymers, mixtures of a depolymerized polymer and a hydrocarbon solvent, and mixtures of a depolymerized polymer, a low-viscosity mineral oil and a hydrocarbon solvent.

[0013] According to this method using a lubricant, however, a certain degree of improvement is achieved but it is still difficult to fill the cavity with the powder sufficiently uniformly or achieve a sufficient degree of compactibility. Among other things, a powder made by a strip casting process or any other rapid quenching process (at a cooling rate of 10^2 °C/s to 10^4 °C /s) has a smaller mean particle size and a sharper particle size distribution than a powder made by an ingot casting process, and therefore, exhibits particularly bad flowability. For that reason, the amount of the powder to fill the cavity may sometimes go beyond its allowable range or the in-cavity fill density may become non-uniform. As a result, the variations in the mass or dimensions of the compacts may exceed their allowable ranges or the compacts may crack or chip.

[0014] As another method for improving the flowability and compactibility of an R-Fe-B based alloy powder, there was a proposal to make a granulated powder.

[0015] For example, Japanese Patent Application Laid-Open Publication No. 63-237402 discloses that the compactibility should be improvable with a granulated powder to be obtained by adding 0.4 mass% to 4.0 mass% of mixture of a paraffin compound (which is liquid at room temperature) and an aliphatic carboxylate to the powder, and mulling and granulating them together. A method in which polyvinyl alcohol (PVA) is used as a granulating agent is also known. It should be noted that the granulating agent, as well as a lubricant, functions as a binder for increasing the strength of the compact.

[0016] If the granulating agent disclosed in Japanese Patent Application Laid-Open Publication No. 63-237402 is used, however, then the binder removability is so bad that the magnetic properties of an R-Fe-B based sintered magnet will be deteriorated by carbon remaining in the sintered body.

[0017] On the other hand, the granulated powder produced by applying a spray dryer method to PVA has high binding force and therefore is too hard to be broken completely even on the application of an external magnetic field. Accordingly, the primary particles thereof cannot be aligned with the magnetic field sufficiently and no magnets with excellent magnetic properties can be obtained. PVA also has bad binder removability and carbon derived from PVA is likely to remain in the magnets. This problem may be overcome by performing a binder removal process within a hydrogen atmosphere. However, it is still difficult to remove that carbon sufficiently.

[0018] To solve the problem that the granulated powder is difficult to break even under the aligning magnetic field, the applicant of the present application proposed a method for making a granulated powder, in which respective powder particles (i.e., primary particles) aligned with a magnetic field applied are coupled together with a granulating agent, by granulating the material powder with a static magnetic field applied thereto (see Japanese Patent Application Laid-Open Publication No. 10-140202). If this granulated powder is used, the magnetic properties are improvable compared with using a granulated powder in which primary particles not aligned with a magnetic field applied are coupled together with a granulating agent. However, it is difficult to align the powder particles being pressed with the magnetic field sufficiently. Consequently, the resultant magnetic properties are lower than a situation where a non-granulated rare-earth alloy powder was used.

[0019] Various granulating agents and granulating methods have been proposed so far as described above. However, a method for mass-producing a rare-earth alloy granulated powder, which has excellent flowability and compactibility and which can contribute to producing magnets with good magnetic properties, has not yet been developed.

[0020] On the other hand, demands for smaller, thinner and performance-enhanced magnets have been escalating. Thus, the development of a method for producing small or thin high-performance magnets with high productivity is awaited. Generally speaking, if a rare-earth alloy sintered body (or a magnet obtained by magnetizing the sintered body) is machined, then its magnetic properties will deteriorate due to a strain caused by the machining process. Such deterioration in magnetic properties is non-negligible in a small magnet. Accordingly, the smaller the size of the magnet to be obtained, the more necessary it is to make a sintered body that has so high dimensional accuracy as to need almost no machining at all and also has the final shape to be obtained. Demands for a rare-earth alloy powder with excellent flowability and compactibility (e.g., an R-Fe-B based alloy powder among other things) have been further growing for these reasons, too.

DISCLOSURE OF INVENTION

[0021] In order to overcome the problems described above, a primary object of the present invention is to provide a method for making a rare-earth alloy granulated powder, which has good flowability and good compactibility and which makes it possible to produce a magnet with excellent magnetic properties, and a method for making a quality rare-earth alloy sintered body with high productivity.

[0022] A method for making a rare-earth alloy granulated powder according to the present invention includes the steps of: (a) preparing a rare-earth alloy powder with remanent magnetization; (b) feeding the powder onto a track, which is defined by a side surface and a lower surface that is sloped so as to decrease its height toward the side surface; and (c) setting up vibrations on the track to give the powder kinetic energy, thereby transporting the powder along the length of the track and granulating the powder under a substantially zero magnetic field by utilizing an agglomeration force produced by the remanent magnetization of the powder and a tumbling action produced by the kinetic energy, whereby the objects described above are achieved.

[0023] In one preferred embodiment, the step (c) includes the step of transporting the powder along the length of the track while making some particles of the powder, located near the side surface, climb the sloped lower surface.

[0024] In another preferred embodiment, the side surface is arranged spirally and is located on the outer side of the track.

[0025] In another preferred embodiment, the step (b) is carried out after the powder has been subjected to particle sizing.

[0026] In another preferred embodiment, the rare-earth alloy is an R-Fe-B based alloy.

[0027] In another preferred embodiment, the powder has a mean particle size of 1.5 μm to 6 μm.

[0028] In another preferred embodiment, a granulated powder with a mean particle size of 0.05 mm to 3.0 mm is obtained.

[0029] A method for making a rare-earth alloy sintered body according to the present invention includes the steps of: making a granulated powder by any of the methods of making a rare-earth alloy granulated powder; filling a cavity with a rare-earth alloy powder, including the granulated powder, without applying a demagnetizing field to the granulated powder; compacting the rare-earth alloy powder including the granulated powder with an aligning magnetic field applied to the alloy powder, thereby making a compact; and sintering the compact.

[0030] An apparatus of making a granulated powder according to the present invention includes: a track, which is defined by a side surface and a lower surface that is sloped so as to decrease its height toward the side surface; a guide surface extending from the side surface, defining the track, toward the center of the track and being tilted in a transporting direction; and a shaker for setting up vibrations on the track.

[0031] In one preferred embodiment, the apparatus further includes a bowl to receive a rare-earth alloy powder with remanent magnetization, and the track is arranged spirally on an inner surface of the bowl.

[0032] In another preferred embodiment, the apparatus further includes: a container for containing a rare-earth alloy material powder; and a magnetizer including a magnetic circuit for applying a magnetic field to the material powder in the container.

[0033] In another preferred embodiment, the apparatus further includes a particle sizer between the magnetizer and the bowl.

BRIEF DESCRIPTION OF DRAWINGS

[0034]

FIG. **1(a)** schematically illustrates the structure of a granulated powder according to a preferred embodiment of the present invention, and FIGS. **1(b)** and **1(c)** schematically illustrate the structures of conventional granulated powders for the purpose of comparison.

FIGS. **2(a)** and **2(b)** illustrate a granulating process step according to a preferred embodiment of the present invention, wherein FIG. **2(a)** is a plan view of a track **22** as viewed from over it, and FIG. **2(b)** is a cross-sectional view thereof as viewed on the plane **B-B'** shown in FIG. **2(a)**.

FIG. **3** schematically illustrates a granulator **120** according to a preferred embodiment of the present invention.

FIG. **4** is a plan view schematically illustrating the configuration of the bowl **120A** of the granulator **120** shown in FIG. **3.**

FIG. **5** is a perspective view partly in section of the bowl **120A** of the granulator **120** shown in FIG. **3.**

FIG. **6** schematically illustrates a granulating system **100** according to a preferred embodiment of the present invention.

BEST MODE FOR CARRYING OUT THE INVENTION

[0035] Hereinafter, a method for making a granulated powder and a method for making a rare-earth alloy sintered body according to preferred embodiments of the present invention will be described with reference to the accompanying drawings. In the following description of preferred embodiments, the features of the present invention will be described as being applied to a method for making a sintered magnet of an R-Fe-B based alloy powder prepared by a strip casting process, which exhibits excellent magnetic properties but low flowability. However, the present invention is in no way limited to those specific preferred embodiments. Thus, a rare-earth alloy powder made by any other method may also be used instead.

[0036] A method for making an R-Fe-B based alloy sintered body according to a preferred embodiment of the present invention includes the steps of: making an R-Fe-B based alloy powder (which will be referred to herein as a "material powder" or "primary particle powder"); generating remanent magnetization in the material powder; granulating the powder by utilizing agglomeration force produced by the remanent magnetization of the material powder; making a compact by pressing the R-Fe-B based alloy powder, including the granulated powder, with a magnetic field applied thereto; and sintering the compact. By magnetizing the resultant sintered body by a known method, an R-Fe-B based sintered magnet can be obtained. It should be noted that the magnetizing process step may be carried out at any arbitrary point in time after the sintering process. For example, the user of the sintered magnet may perform the magnetizing process step just before he or she uses the sintered magnet. Even a non-magnetized one will also be referred to herein as a "sintered magnet".

[0037] In a method for making an R-Fe-B based alloy sintered body according to a preferred embodiment of the present invention, the powder is granulated by utilizing the agglomeration force produced by the remanent magnetization of the material powder. Accordingly, it is possible to either reduce the amount of a granulating agent to be added or use a binder with low binding force than a conventional one. Furthermore, even the addition of the granulating agent itself may be omitted.

[0038] Hereinafter, the features of a granulated powder making method and a resultant granulated powder according to a preferred embodiment of the present invention will be described with reference to FIGS. **1(a), 1(b)** and **1(c).** On the left-hand side of FIG. **1,** illustrated schematically are the structures of respective granulated powders. On the righthand side of FIG. **1,** illustrated schematically are the states of respective granulated powders to which an aligning magnetic field has been applied in a cavity for the purpose of compaction. More specifically, FIG. **1(a)** illustrates a granulated powder **12a** according to a preferred embodiment of the present invention, FIG. **1(b)** illustrates a conventional granulated powder **12b** for which a granulating agent has been used, and FIG. **1(c)** illustrates a granulated powder **12c** obtained by the method described in Japanese Patent Application Laid-Open Publication No. 10-140202 identified above.

[0039] As shown in FIG. **1(a)**, in the granulated powder **12a** of this preferred embodiment, primary particles 10a with remanent magnetization are weakly coupled together via magnetic agglomeration force. In the illustrated example, no granulating agent is supposed to be used. These primary particles **10a** with remanent magnetization are magnetically coupled together so as to form a magnetic closed circuit, and the remanent magnetization of the granulated powder **12a** is very small (e.g., more than about 0 mT and equal to or smaller than about 10 mT (millitesla)). In this granulated powder **12a**, the remanent magnetization of the primary particles **10a** is oriented at random unlike the granulated powder **12c** shown in FIG. **1(c).** The primary particles **10a** may have a mean particle size of about 1.5 μm to about 6.0 μm and the granulated powder **12a** may have a mean particle size of about 0.05 mm to about 3.0 mm, for example. The remanent magnetization may be measured by inserting a probe of a gauss meter into the granulated powder.

[0040] This granulated powder 12a has a moderate particle size and an appropriately shape and can exhibit excellent flowability. In addition, this granulated powder 12a also has low remanent magnetization and can be loaded into a cavity easily and uniformly without causing any bridging. Furthermore, these primary particles **10a** are just coupled together via the magnetic agglomeration force. Accordingly, as shown on the left-hand side of FIG. **1(a)**, the granulated powder **12a** can be broken down into the primary particles **10a** just as intended by applying an aligning magnetic field (of about 0.1 T to about 0.8 T, for example) thereto. As a result, the primary particles **10a** can be aligned with the magnetic field applied. Also, since the granulated powder **12a** includes no granulating agent, the amount of carbon included in the sintered body never increases. A magnet obtained by magnetizing a sintered body made from this granulated powder **12a** has substantially the same magnetic properties as a magnet obtained without granulating the material powder (with substantially zero remanent magnetization) at all. That is to say, by using the granulated powder of the preferred embodiment of the present invention, the flowability and compactibility can be improved without deteriorating the magnetic properties. Optionally, it is naturally possible to add a granulating agent for the purpose of increasing the strength of the compact, for example. As such a granulating agent is used just as an additional agent, the granulating agent does not have to exhibit strong binding force. Thus, the amount and type of the granulating agent may be selected so as not to deteriorate the magnetic properties.

[0041] In contrast, the granulated powder 12b, obtained by binding the primary particles **10b** of the material powder together with a granulating agent **14,** cannot be sufficiently broken down even under an aligning magnetic field as shown in FIG. **1(b).** As a result, the magnetic properties of the resultant sintered magnet deteriorate. Compared with a magnet obtained without granulating the material powder at all, the remanent magnetization of that sintered magnet decreases by about 1% to about 10%. It should be noted that arrows are omitted from the primary particles **10b** of the granulated powder **12b** shown in FIG. **1(b)** because the particles 10b have no remanent magnetization.

[0042] Furthermore, if a granulated powder **12c** is obtained by binding and fixing primary particles **10c** together with a granulating agent **14** while aligning the primary particles **10c** under a static magnetic field as shown in FIG. **1(c)**, then the deterioration in magnetic properties can be minimized but the granulated powder **12c** cannot be fully broken down into the primary particles **10c.** Accordingly, compared with a magnet obtained without granulating the material powder at all, the remanent magnetization of the resultant sintered magnet decreases by about one to several percent. Also, as schematically illustrated in FIG. **1(c),** the granulated powder **12c** is elongated in the directions of the magnetic poles, which is disadvantageous in terms of flowability. Furthermore, since the granulated powder **12c** has relatively large remanent magnetization, the granulated powder **12c** will produce bridging and cannot be loaded into a cavity unless demagnetized once.

[0043] In contrast, the granulated powder **12a** of the preferred embodiment of the present invention is almost spherical in shape, has too small remanent magnetization to require any demagnetization, and can fill a cavity easily and uniformly. Accordingly, a so-called "measuring and loading technique", in which a predetermined mass of granulated powder is measured in advance and then loaded into a cavity, can be adopted. As described above, the granulated powder 12a of the preferred embodiment of the present invention can exhibit excellent flowability and cavity-filling ability and can contribute to making a sintered magnet substantially without deteriorating the magnetic properties.

[0044] A granulated powder according to a preferred embodiment of the present invention is obtained by a granulating method including the steps of giving kinetic energy to particles of a material powder with remanent magnetization and allowing the particles to grow under a tumbling action produced by the kinetic energy given. Optionally, a granulating agent may be added if necessary.

[0045] In a method for making a granulated powder according to a preferred embodiment of the present invention, the step of generating remanent magnetization in the material powder may be carried out at an arbitrary point in time before the material powder is fed onto the bottom of the apparatus of making the granulated powder. However, the primary particles **10a** of the granulated powder **12a** of this preferred embodiment are just coupled together under magnetic agglomeration force produced by the remanent magnetization. Accordingly, the granulated powder **12a** is broken down upon the application of an external magnetic field. For that reason, the particles are allowed to grow under a substantially zero magnetic field. This is contrary to the method for making the granulated powder **12c** shown in FIG. **1(c)** in which a magnetic field needs to be applied continuously to align the primary particles **10c** until the granulated powder **12** is definitely fixed with the granulating agent **14.** As used herein, the "substantially zero magnetic field" refers to a magnetic field which is weak enough to obtain a granulated powder where a magnetic closed circuit has been formed by the remanent magnetization of the powder and to have no effects on the remanent magnetization of the powder while the particles grow due to the tumbling action.

[0046] The magnetic field to be applied to generate remanent magnetization may be any of various magnetic fields. Since the primary particles may have a little remanent magnetization, an alternating demagnetizing field is preferably used. However, the magnetic field to generate remanent magnetization does not have to be such an alternating demagnetizing field but may also be a monotonically demagnetizing field, another type of pulse magnetic field or a static magnetic field.

[0047] It should be noted that even after the remanent magnetization has been generated, a material powder with low coercivity might lose the magnetization and the shape of the granulated powder before the final granulated powder is obtained. For that reason, the material powder preferably has relatively high coercivity. Specifically, if the coercivity value of a material powder, which has been loaded into a container so as to have a tap density of 2.0 g/cm^3, is measured by a BH tracer as the apparent coercivity of the material powder, the material powder preferably has a coercivity of at least 60 kA/m, more preferably 70 kA/m or more. For example, an R-Fe-B based alloy preferably includes at least 1.2 mass% of Dy, at least 1 mass% of Tb or at least 1 mass% of Dy and Tb combined.

[0048] Considering the flowability and compactibility, the R-Fe-B based alloy powder to be pressed and compacted preferably consists of only the granulated powder prepared as described above. Alternatively, a mixture of the granulated powder and the material powder (i.e., powder of primary particles) may also be used. However, as the percentage of the material powder increases, the flowability decreases. Accordingly, to improve the flowability sufficiently effectively by the granulating technique, the alloy powder preferably consists essentially of the granulated powder alone. Also, when the mixture of the material powder and the granulated powder is used, the surface of the material powder particles is preferably coated with a lubricant. By coating the surface of the primary particles with a lubricant, the flowability of the R-Fe-B based powder can be improved and the oxidation of the R-Fe-B based alloy can be prevented as well. Furthermore, in pressing the powder under a magnetic field, the powder particles can also be aligned more easily. It should be noted that not only a powder consisting essentially of a rare-earth alloy alone (possibly with a surface oxide layer) but also a powder, including a granulating agent and/or a lubricant as well as the rare-earth alloy powder and being subjected to the compaction process, will be referred to herein as "rare-earth alloy powders".

[0049] Hereinafter, a method for making a magnet from an R-Fe-B based alloy sintered body according to a preferred embodiment of the present invention will be described step by step.

[0050] First, flakes of an R-Fe-B based alloy are made by a strip casting process (see United States Patent No. 5,383,978, for example). Specifically, an R-Fe-B based alloy, prepared by a known method, is melted by an induction heating process to obtain a molten alloy. The R-Fe-B based alloy may also have the composition disclosed in United States Patent No. 4,770,723 or No. 4,792,368. In a typical composition of the R-Fe-B based rare-earth alloy, Nd or Pr is usually used as R, a portion of Fe may be replaced with a transition element (e.g., Co), and a portion of B may be replaced with C.

[0051] This molten alloy is maintained at 1,350 °C and then rapidly quenched on a single roller under the conditions including a roller peripheral velocity of about 1 m/s, a cooling rate of 500 °C /s and an undercooling of 200 °C, thereby obtaining alloy flakes with a thickness of 0.3 mm. By decrepitating these alloy flakes by a hydrogen absorption process, an alloy coarse powder is obtained. Then, this alloy coarse powder is finely pulverized by a jet mill within a nitrogen gas atmosphere, thereby obtaining an alloy powder (i.e., material powder) with a mean particle size of 1.5 μm to 6 μm and a specific surface area of about 0.45 m^2/g to about 0.55 m^2/g as measured by a BET method. This material powder has a true density of 7.5 g/cm^3.

[0052] Next, remanent magnetization is generated in the material powder obtained in this manner. In this example, an alternating demagnetizing field with a peak magnetic field of 1.0 T is applied thereto by a magnetizer.

[0053] Subsequently, the material powder with remanent magnetization is granulated. The applicant of the present application described a method for granulating a material powder with remanent magnetization by a fluid-bed granulating technique in Japanese Patent Applications No. 2001-362436 and No. 2002-298621. In the preferred embodiment of the present invention, however, a granulated powder can be obtained by a shaking granulating technique more easily than by the method described in the previous applications.

[0054] A method for making a granulated powder according to this preferred embodiment of the present invention includes the steps of: (a) preparing a rare-earth alloy powder with remanent magnetization; (b) feeding the powder onto a track, which is defined by a side surface and a lower surface that is sloped so as to decrease its height toward the side surface; and (c) setting up vibrations on the track to give the powder kinetic energy, thereby transporting the powder along the length of the track and granulating the powder under a substantially zero magnetic field by utilizing an agglomeration force produced by the remanent magnetization of the powder and a tumbling action produced by the kinetic energy. The step (c) preferably further includes the step of transporting the powder along the length of the track while making some particles of the powder, located near the side surface, climb the sloped lower surface.

[0055] This granulating process step will be described with reference to FIGS. **2(a)** and **2(b).** FIG. **2(a)** is plan view of a track **22** as viewed from over it, and FIG. **2(b)** is a cross-sectional view thereof as viewed on the plane **B-B'** shown in FIG. **2(a).**

[0056] The material powder with remanent magnetization is granulated while being transported along the track **22** from left to right in FIG. **2(a).** As shown in FIG. **2(b),** the track **22** is defined by a side surface **22a** and a sloped lower surface **22b** that decreases its height toward the side surface **22a.** In this preferred embodiment, a configuration in which the track **22** is arranged spirally on the inner surface of a bowl is illustrated as an example as will be described in detail later. The side surface **22a** is located on one side of the track **22** (i.e., on the outer side of the spiral track) while the lower surface **22b** is tilted toward one direction. However, if a linearly extending track is used, for example, its cross-sectional structure may have the structure shown in FIG. **2(b)** on the left-hand side, too, so as to be symmetric with respect to the transporting direction (i.e., the direction in which the track extends). Nevertheless, when the track **22** is arranged spirally, a relatively long track **22** can be defined within a rather small area.

[0057] As pointed by the arrows in FIG. **2(b),** the track **22** is shaken both horizontally and vertically. The granulation is done by utilizing the tumbling action produced by the kinetic energy that has been given to the powder by these vibrations and the agglomeration force produced by the remanent magnetization of the powder. The tumbling action is produced mainly by the horizontal vibrations. However, the powder is preferably shaken both horizontally and vertically because the density of the powder can be increased effectively by the vertical vibrations. In addition, the amplitude of the horizontal vibrations also has an effect on the transportation rate. That is to say, the transportation rate can be increased by widening the amplitude of the horizontal vibrations.

[0058] The amplitudes and frequencies of the horizontal and vertical vibrations, as well as the track length, are appropriately defined with the efficiency of granulation and the transportation rate taken into consideration. From the standpoint of granulation efficiency, the vertical vibrations preferably have an amplitude of at least 0.2 mm, more preferably 0.3 mm or more. The horizontal vibrations preferably have an amplitude of at least 0.5 mm, more preferably 1.0 mm or more, in view of the transportation rate. However, if the amplitude exceeded 2.0 mm, sufficient granulation effects could not be achieved and the flowability might not increase so much as expected. The frequencies of the horizontal and vertical vibrations may fall within the range of 70 Hz to 80 Hz but are not particularly limited. The phase relationship between the horizontal and vertical vibrations is appropriately defined and may be defined so as to achieve elliptical vibrations.

[0059] The track length is preferably no shorter than 4,000 mm. However, if an apparatus with a short track length is used, substantially the same effects as those obtained by extending the track length can be achieved by performing the granulating process step a number of times. The track length has an effect on the particle size and shape of the granulated powder. Specifically, if the track length were too short, then a sufficiently big granulated powder could not be obtained, the shape of the granulated powder might not be regular enough, and/or the percentage of sufficiently big granulated powder particles might be low.

[0060] On the vibrating track **22,** relatively small granulated powder particles **1a** gather toward the higher-level portion of the sloped lower surface **22b,** while relatively big granulated powder particles **1b** gather toward the lower-level portion of the sloped lower surface **22b** (i.e., near the side surface **22a).** If the granulated powder is unevenly distributed in this manner according to their sizes, then the granulation efficiency will decrease. Thus, by providing a guide surface **22c,** which extends from the side surface **22a** toward the center of the track **22** and is tilted in the transporting direction as shown in FIG. **2(a),** the relatively big granulated powder particles **1b,** gathering toward the side surface **22a** of the track **22,** can be moved upward (i.e., toward the higher-level portion of the lower surface **22b)** against the slope of the lower surface **22b.** Then, the relatively big granulated powder particles **1b** and the relatively small granulated powder particles **1a** will be blended together, thus achieving the granulation highly efficiently. The guide surface **22c** preferably defines a tilt angle of 30 degrees to 60 degrees with respect to the transporting direction (i.e., the angle defined by a normal to the guide surface **22c** with respect to the transporting direction is preferably 120 degrees to 150 degrees). As used herein, the "transporting direction" refers to the length direction of the track or a tangential direction of the track if the track is winding. The reasons are as follows. Specifically, if the tilt angle of the guide surface **22c** were less than 30 degrees, then the granulation effect would be so insufficient as to increase the percentage of small granulated powder particles and the variation in particle size significantly. However, if the tilt angle exceeded 60 degrees, then the efficiency of transportation would decrease, which is an unwanted scenario.

[0061] The interval between adjacent guide surfaces **22c** is appropriately defined according to a combination of the width and length of the track **22** and the transportation rate (i.e., a combination of vibration conditions). The interval between adjacent guide surfaces **22c** may be defined at about 80 mm or more. This is because if the interval were shorter than about 80 mm, then the granulation effect would decrease, which is not advantageous. However, if the interval exceeded about 200 mm, then the granulation effect produced by the guide surfaces would decline, which is not beneficial, either.

[0062] Next, an apparatus **120** for making a granulated powder (which will be referred to herein as a "granulator") according to a preferred embodiment of the present invention will be described with reference to FIGS. **3** through **5.**

[0063] As shown in FIG. **3,** the granulator **120** includes a bowl **120A** and a shaker **120B.** The shaker **120B** may be substantially the same as a known bowl vibrating parts feeder (produced by Shinko Electric Co., Ltd., for example). Thus, the description of the configuration of the shaker **120B** (see Japanese Patent Application Laid-Open Publication No. 2001-114412, for example) will be omitted herein. Instead, the structure of the bowl **120A** will be described below.

[0064] FIG. **4** schematically illustrates the configuration of the bowl **120A** as viewed from over it, and FIG. **5** is a perspective view partly in section of the bowl **120A.**

[0065] On the inner surface of the bowl **120A,** a track **122,** which is defined by a side surface **122a** and a sloped lower surface **122b** that decreases its height toward the side surface 122a, is arranged spirally. Also, guide surfaces, which extend from the side surface **122a** toward the center of the track **122** and which are tilted in the transporting direction (i.e., corresponding to the guide surfaces **22c** shown in FIG. **2),** are defined by the side surfaces of convex portions **122d.** It should be noted that each of those convex portions **122d** includes not only the surface tilted in the transporting direction and functioning as the guide surface (i.e., the guide surface **22c** shown in FIG. **2)** and a surface tilted in the opposite direction. Due to the presence of that surface tilted in the opposite direction, it is possible to prevent the powder from being collected behind the guide surface, thus improving the efficiency of transportation. Naturally, a baffle with the guide surface **22c** such as that shown in FIG. **2** may be used instead of the convex portion **122d.**

[0066] Meanwhile, baffles **122c,** which extend from the inner side of the track **122** (i.e., the higher-level portion of the lower surface **122b)** toward the center of the track **122** and which are tilted in the transporting direction, are further provided. These baffles **122c** work so as to push the granulated powder, which has been moved by the guide surface of the convex portion **122d** upward against the slope of the lower surface **122b,** back to the side surface **122a** (i.e., toward the lower-level portion of the lower surface **122b)** again. By providing these baffles **122c,** the granulation effects can be increased and the granulated powder can be flushed more efficiently. The baffles **122c** are preferably provided for the inside portion of the spiral track **122.** In the arrangement shown in FIG. **4,** the baffles **122c** are provided for just 1.5 inner rounds of approximately 3 rounds of track **122.**

[0067] The bowl **120A** as a whole has a mortar shape and a rare-earth alloy powder is fed into the bottom **124** at the center of the bowl **120A.** A conical protrusion **125** is provided at the center of the bottom **124** and the circular bottom thereof is surrounded with ridge-shaped small protrusions **126,** which extend in the tangential directions of the circular bottom, thereby supplying the fed powder onto the track **122** on the inner surface efficiently. To improve the granulation efficiency, the material powder is preferably subjected to particle sizing before being fed into the bowl **120A.**

[0068] The powder that has been fed into the bowl **120A** is granulated as already described with reference to FIG. 2, while climbing the inner surface of the mortar bowl **120A** from the bottom thereof and along the spiral track **122,** and then transported to an outlet port **128** at the top of the bowl **120A.** The outlet port **128** may be connected to a feeder (not shown) for use in the next compacting process step.

[0069] In this case, if the contact resistance (frictional resistance) between the powder with remanent magnetization and the surfaces of the track **122** (i.e., the side surface **122a** and the lower surface **122b)** or the surfaces of the baffles **122c** and convex portions **122d** were too high, then the powder would adhere to those surfaces to possibly decrease the granulation efficiency. Accordingly, those surfaces to contact with the powder are preferably smooth. For that reason, the bowl **120A** is preferably made of a stainless steel such as mirror-polished SUS and its surface is preferably further coated with urethane. Furthermore, if a granulating agent were added to the powder, then the powder would adhere to the surfaces of the bowl **120A** easily. Thus, the granulating agent would rather not be used in many cases. It should be noted that even if a granulating agent was added to a powder with no remanent magnetization, it was difficult to obtain a granulated powder by this method.

[0070] Exemplary specifications of the granulator **120** of this preferred embodiment may be as follows:

- Vertical vibrations: amplitude of 0.3 mm
- Horizontal vibrations: amplitude of 1.5 mm
- Bottom **124** of bowl: diameter of 350 mm
  Tilt angles to horizontal plane:
  8 degrees on bottom, 10 degrees on track
- Protrusion **125:** bottom diameter of 100 mm height of 50 mm
- Track **122:** width of 35 mm, side surface height of 30 mm, total length of 4,000 mm
- Diameter of outermost track: 560 mm
- Baffles **122c:** angle defined by the side surface with respect to transporting direction is 45 degrees, side surface length of 22 mm
- Convex portions **122d:** angle defined by the side surface with respect to transporting direction is 45 degrees, side surface length of 30 mm

[0071] According to the granulating method of the present invention, there is no need to control the pressure of the gas surrounding the powder, and therefore, the granulating process step may be carried out at the atmospheric pressure. However, since the rare-earth alloy powder is easily oxidizable, the granulating process step is preferably carried out within an inert gas (e.g., nitrogen or rare gas) atmosphere. For example, the overall granulator **120** may be covered with a shield that is filled with a nitrogen gas. The shield does not have to have an airtight structure but may be ventilated with the nitrogen gas, for example.

[0072] A granulated powder made from the rare-earth alloy powder described above (having a mean particle size of 1.5 μm to 6 μm) preferably has a mean particle size of 0.05 mm to 3.0 mm. Generally speaking, very few primary particles are included in a granulated powder and the number of tertiary or even higher- order particles contained there is also very small. For that reason, the mean particle size of secondary particles may be regarded as substantially representing that of the granulated powder. In this preferred embodiment, the mean particle size of secondary particles, obtained by observing the powder with a microscope, is used as the mean particle size of the granulated powder. If the mean particle size of the granulated powder were less than 0.05 mm, then the flowability could not be improved significantly and it would be difficult to obtain a uniform compact with a sufficient density. However, if the mean particle size of the granulated powder exceeded 3 mm, then the cavity fill density would decrease and it should be difficult to obtain a uniform compact with a sufficient density, too. More preferably, the mean particle size of the granulated powder falls within the range of 0.1 mm to 1.5 mm. By using the granulator **120** illustrated herein, a granulated powder with a mean particle size falling within that range of 0.1 mm to 1.5 mm can be obtained efficiently.

[0073] Next, the process step of pressing and compacting the resultant granulated powder is carried out. Before describing it, another advantage of the granulator **120** will be described. Unlike a conventional fluid-bed granulator, this granulator **120** can simplify and/or automate the production line of rare-earth alloy compacts for sintered magnets. That is to say, a granulating system **100,** including the granulator **120** described above, can be fabricated as schematically shown in FIG. **6.**

[0074] In the granulating system **100,** the granulator **120** described above and a number of machines for performing preprocessing for the granulator **120** are connected together vertically. More specifically, in this granulating system **100,** a hopper **130** to receive the material powder, a meter (scale) **140,** a magnetizer **150,** and a particle sizer **160** are connected together with coupling pipes.

[0075] The rare-earth alloy material powder is fed into the hopper **130,** is measured by the meter **140** to a predetermined mass, and then supplied to the container (with the predetermined mass) of the magnetizer **150.** The magnetizer **150** includes a magnetic circuit (not shown). When a predetermined pulse current is supplied to the coil, the magnetizer **150** generates an alternating demagnetizing field, thereby applying remanent magnetization to the material powder in the container.

[0076] The powder with remanent magnetization is subjected to particle sizing by the particle sizer **160** so as to consist of blocks of a predetermined size, which are then supplied to the bowl **120A** of the granulator **120.** The particle sizer **160** may be implemented as a mesh (of wires) with an aperture size of 0.5 mm to 1.5 mm. The powder, having been sorted into blocks of the predetermined size by the particle sizer **160** and then fed to the granulator **120,** is granulated with those blocks used as nuclei. Thus, the particle size range of the resultant granulated powder will be limited and no abnormally big granulated powder particles will be created. The aperture size of the mesh may be appropriately defined according to the target particle size of the granulated powder. In this preferred embodiment, however, since the agglomeration force produced by the remanent magnetization is utilized, the particle size of the resultant granulated powder is restricted by the magnitude of the remanent magnetization. Thus, once the aperture size falls out of that range, the effect of the particle sizing would decrease. Particularly if the aperture size were less than 0.5 mm, then it would be hard for those blocks to function as the nuclei of granulation, and therefore, the granulation efficiency would decrease.

[0077] Optionally, the mesh may be folded like a bellows, for example. By folding the mesh, the powder sorting processing can be performed more efficiently. To further increase the efficiency of the powder sorting processing, the mesh may be vibrated by connecting the mesh to a vibrating mechanism, for example.

[0078] The granulated powder is flushed through the outlet port **128** (see FIG. **4)** of the granulator **120.** If this granulated powder is supplied by a feeder, for example, to a feeder box for use in the next compacting process step, then the manufacturing process can be fully automated from the material powder feeding process step through the pressing/compacting process step.

[0079] Next, the resultant granulated powder is pressed and compacted, thereby making compacts. In this preferred embodiment, the compacts are made of only the granulated powder. This pressing/compacting process step may be carried out with a known press machine. Typically, a uniaxial press machine for pressing a powder in a die cavity (also called a "die hole") with upper and lower punches is used.

[0080] Then, the die cavity of the uniaxial press machine is filled with the granulated powder. This process step of filling the cavity with the granulated powder may be carried out by either a filling method using a sieve or a filling method using a feeder box as disclosed in Japanese Patent Gazette for Opposition No. 59-40560, Japanese Patent Application Laid-Open Publication No. 10-58198, Japanese Utility Model Application Laid-Open Publication No. 63-110521 and Japanese Patent Application Laid-Open Publication No. 2000-248301. These two methods are sometimes called "dropping methods" collectively.

[0081] Particularly, in making a small compact, the granulated powder is preferably measured with the cavity to an amount corresponding to the internal volume of the cavity. For example, a feeder box having an opening at the bottom may be shifted to over the cavity to let the granulated powder drop due to its own gravity (i.e., by itself), and then the excess of the granulated powder loaded into the cavity is sliced off. In this manner, a predetermined amount of granulated powder can be loaded relatively uniformly. It is naturally possible to fill the cavity with a separately measured granulated powder using a funnel, for example.

[0082] After the cavity has been filled with the granulated powder, the upper punch of the uniaxial press machine is lowered. With the cavity closed in this manner, an aligning magnetic field is applied to the powder, thereby breaking down the granulated powder into primary particles and also aligning those primary particles with the magnetic field applied. The granulated powder of this preferred embodiment of the present invention can be broken down into primary particles just as intended with the application of a relatively weak magnetic field of 0.1 T to 0.8 T. However, to achieve a sufficient degree of magnetic alignment, the magnetic field applied preferably has a strength of about 0.5 T to about 1.5 T. The magnetic field direction may be perpendicular to the pressing direction, for example. While the magnetic field is applied in this manner, the powder is pressed uniaxially between the upper and lower punches at a pressure of 98 MPa, for example. As a result, a compact with a relative density (i.e., the ratio of the compact density to the true density) of 0.5 to 0.6 can be obtained. If necessary, the magnetic field direction may be parallel to the pressing direction. The granulated powder obtained by the process of the present invention has an adequate strength, i.e., too strong to be broken in the filling process step but weak enough to be broken down into primary particles with the application of the aligning magnetic field.

[0083] Next, the resultant compact is sintered at a temperature of about 1,000 °C to about 1,180 °C for approximately one to six hours within either a vacuum or an inert gas atmosphere. The granulated powder of this preferred embodiment includes either no granulating agent at all or just a small amount of granulating agent, if any, which is small enough to be substantially eliminated by the sintering process. Thus, there is no need to separately provide any binder removal process. It should be noted that a typical conventional binder removal process is carried out at a temperature of about 200 °C to about 800 °C for approximately three to six hours within an inert gas atmosphere at a pressure of about 2 Pa.

[0084] Subsequently, by subjecting the resultant sintered body to an aging treatment at a temperature of about 450 °C to about 800 °C for approximately one to eight hours, an R-Fe-B based sintered magnet can be obtained. Thereafter, the magnet is magnetized at an arbitrary stage, thereby completing an R-Fe-B based sintered magnet.

[0085] According to the present invention, a granulated powder with excellent flowability and compactibility is used as described above. Thus, the cavities can be filled with such a granulated powder uniformly with the variation in fill density reduced. Accordingly, compacts obtained by the compaction process have a reduced variation in mass or size. Furthermore, those compacts rarely crack or chip.

[0086] In addition, the primary particles of the granulated powder of this preferred embodiment are just coupled together substantially due to the magnetic agglomeration force produced by the remanent magnetization. Thus, by applying an aligning magnetic field thereto, the powder can be broken down into the primary particles just as intended. Accordingly, the degree of magnetic alignment of the primary particles never drops. Furthermore, the deterioration in magnetic properties, which would otherwise be caused if the carbon atoms of a granulating agent remained in the sintered body, can be minimized, too. Consequently, a sintered magnet with excellent magnetic properties can be obtained. Thus, according to the present invention, R-Fe-B based alloy sintered magnets of quality can be manufactured with high productivity.

Examples

[0087] Hereinafter, specific examples of the present invention will be described.

[0088] An R-Fe-B based alloy powder was made in the following manner. A molten alloy was prepared by using ferroboron alloy including electrolytic iron with a purity of 99.9% and 19.8 mass% of B, and Nd and Dy with purity of 99.7% or more as respective start materials. Flakes of an R-Fe-B based alloy, having a composition including 34.0 mass% of Nd, 1.0 mass% of Dy, 1.0 mass% of B and Fe as the balance, and flakes of another R-Fe-B based alloy, having a composition including 30.0 mass% of Nd, 5.0 mass% of Dy, 1.0 mass% of B and Fe as the balance, were obtained as Examples Nos. 1 and 2, respectively, from this molten alloy by a strip casting process. These alloy flakes were finely pulverized by using a jet mill within an inert gas (e.g., N_2 gas with a gas pressure of 58.8 MPa), thereby making a material powder with a mean particle size of 3 μm. The powders representing Examples Nos. 1 and 2 had coercivities of 60 kA/m and 120 kA/m, respectively.

[0089] Next, remanent magnetization was generated in the material powders of these specific examples by applying an alternating demagnetizing field (with a peak magnetic field of 1.0 T) thereto. Thereafter, granulated powders with a mean particle size of 0.3 mm were obtained by using the granulator 120 described above without adding any granulating agent. Each of the granulated powders thus obtained had a remanent magnetization of about 0.2 mT. The rest angles measured for the respective granulated powders are shown in the following Table 1. Another granulated powder was prepared as Comparative Example No. 1 by a tumbling granulating technique with no remanent magnetization generated in the material powder of Example No. 1 and with 2 mass% of isoparaffin used as a granulating agent. The rest angle of that granulated powder, along with the rest angle of the material powder of Example No. 1 itself, is also shown in the following Table 1:

Table 1

Powder to press	Granulated?	Remanent Magnetization	Granulating Agent	Rest angle
Example 1	Yes	Yes	Not added	42 degrees
Example 2	Yes	Yes	Not added	41 degrees
Comp. Ex. 1	Yes	No	Added	44 degrees
Comp. Ex. 2	No	No	Not added	About 52 degrees

[0090] A powder with a large rest angle has bad flowability. Thus, the smaller the rest angle, the higher the flowability. In Comparative Example No. 2, unless the material powder was granulated, the rest angle was as large as about 52 degrees and the flowability was bad. In contrast, in each of Examples Nos. 1 and 2 and Comparative Example Nos. 1 in which the powders were granulated, the rest angle decreased to less than 45 degrees. Among other things, the granulated powders representing Examples Nos. 1 and 2 had smaller rest angles and exhibited better flowability than the powder to be pressed representing Comparative Example No. 1. That is to say, it can be seen that by taking advantage of remanent magnetization, the flowability can be improved even without using any granulating agent.

[0091] Each of the powders to be pressed shown in Table 1 was loaded into a cavity with a length of 20 mm, a width of 15 mm and a depth of 10 mm by the method using a feeder box as described above and then pressed and compacted uniaxially (under a pressure of 98 MPa and with an aligning magnetic field of 1.3 T applied perpendicularly to the pressing direction). These loading and compacting process steps were carried out under the same conditions for all of the examples of the present invention and comparative examples. It should be noted that compacts with various compact densities (i.e., green densities) were obtained with the pressing conditions changed.

[0092] Each of the resultant compacts was sintered at 1,060 °C for approximately four hours within an Ar atmosphere, and then subjected to an aging treatment at 500 °C for one hour, thereby obtaining a sintered body. Thereafter, this sintered body was further magnetized at 2,387 kA/m to obtain a sintered magnet. 50 samples were obtained for each of the examples of the present invention and comparative examples. The remanences B_r (T) of the resultant sintered magnets are shown in the following Table 2:

Table 2

	Br (T)
Example 1	1.36
Example 2	1.27
Comparative example 1	1.33
Comparative example 2	1.36

[0093] As can be seen from Table 2, substantially no difference was sensible between Br of Example No. 1 and Br of Comparative Example No. 2. Thus, the sintered magnet exhibited excellent magnetic properties. Br of Comparative Example No. 1 to which a granulating agent was added was lower than Br of Example No. 1 or Comparative Example No. 2. This is because the granulating agent remained as carbon in the sintered magnet.

[0094] As described above, by making a granulated powder by utilizing the magnetic agglomeration force produced by the remanent magnetization of primary particles, even if no granulating agent is used, at least the same degree of flowability is achieved compared with a conventional granulated powder to which a granulating agent is added. Accordingly, a sintered magnet exhibiting better magnetic properties can be produced with at least similar productivity compared with the conventional one. Furthermore, if a granulated powder is produced by utilizing only the remanent magnetization of primary particles, deterioration in magnetic properties can be substantially eliminated.

INDUSTRIAL APPLICABILITY

[0095] The present invention provides a method for making a rare-earth alloy granulated powder, which has good flowability and good compactibility and which makes it possible to produce a magnet with excellent magnetic properties. A method for making a high quality rare-earth alloy sintered body with high productivity by using such a granulated powder is provided.

[0096] According to the present invention, the flowability and compactibility of a rare-earth alloy powder can be improved without deteriorating the magnetic properties. Thus, even a sintered magnet, which should have too intricate a shape to be pressed and compacted easily and which should have sacrificed its magnetic properties to a certain degree in the prior art, can also have improved magnetic properties. In addition, the granulating time can be shortened and the binder removal process can be omitted. As a result, the productivity of rare-earth sintered magnets can be increased.

Claims

1. A method for making a rare-earth alloy granulated powder, the method comprising the steps of:

(a) preparing a rare-earth alloy powder with remanent magnetization;
(b) feeding the powder onto a track, which is defined by a side surface and a lower surface that is sloped so as to decrease its height toward the side surface; and
(c) setting up vibrations on the track to give the powder kinetic energy, thereby transporting the powder along the length of the track and granulating the powder under a substantially zero magnetic field by utilizing an agglomeration force produced by the remanent magnetization of the powder and a tumbling action produced

by the kinetic energy.

2. The method of claim 1, wherein the step (c) includes the step of transporting the powder along the length of the track while making some particles of the powder, located near the side surface, climb the sloped lower surface.

3. The method of claim 1 or 2, wherein the side surface is arranged spirally and is located on the outer side of the track.

4. The method of one of claims 1 to 3, wherein the step (b) is carried out after the powder has been subjected to particle sizing.

5. The method of one of claims 1 to 4, wherein the rare-earth alloy is an R-Fe-B based alloy.

6. The method of one of claims 1 to 5, wherein the powder has a mean particle size of 1.5 μm to 6 μm.

7. The method of one of claims 1 to 6, wherein a granulated powder with a mean particle size of 0.05 mm to 3.0 mm is obtained.

8. A method for making a rare-earth alloy sintered body, the method comprising the steps of:

making a granulated powder by the method of one of claims 1 to 7;
filling a cavity with a rare-earth alloy powder, including the granulated powder, without applying a demagnetizing field to the granulated powder;
compacting the rare-earth alloy powder including the granulated powder with an aligning magnetic field applied to the alloy powder, thereby making a compact; and
sintering the compact.

9. An apparatus for making a granulated powder, the apparatus comprising:

a track, which is defined by a side surface and a lower surface that is sloped so as to decrease its height toward the side surface;
a guide surface extending from the side surface, defining the track, toward the center of the track, the guide surface being tilted in a transporting direction; and
a shaker for setting up vibrations on the track.

10. The apparatus of claim 9, further comprising a bowl to receive a rare-earth alloy powder with remanent magnetization, wherein the track is arranged spirally on an inner surface of the bowl.

11. The apparatus of claim 10, further comprising: a container for containing a rare-earth alloy material powder; and a magnetizer including a magnetic circuit for applying a magnetic field to the material powder in the container.

12. The apparatus of claim 11, further comprising a particle sizer between the magnetizer and the bowl.

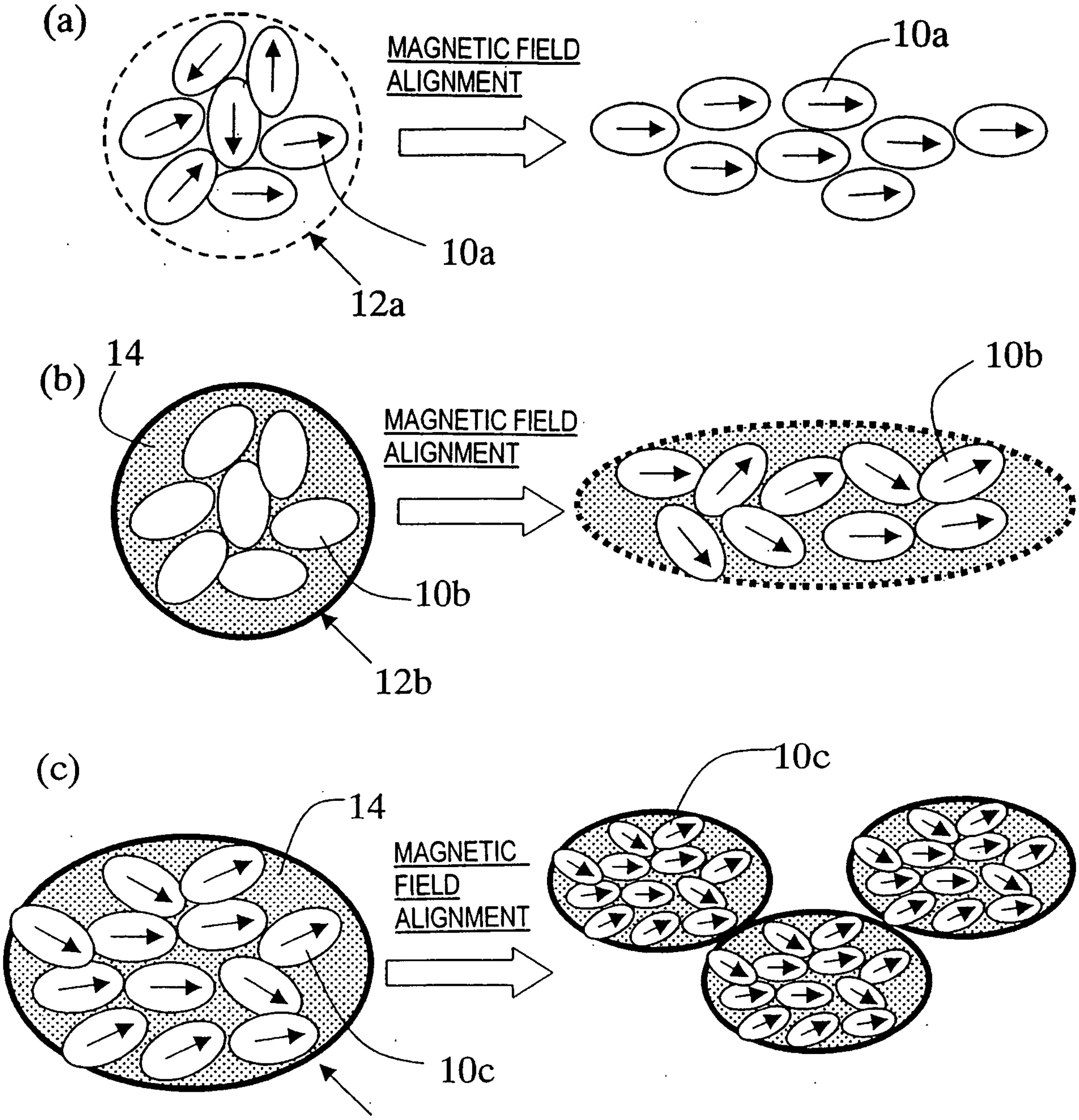

(a)
MAGNETIC FIELD ALIGNMENT
10a
10a
12a
(b)
14
MAGNETIC FIELD ALIGNMENT
10b
10b
12b
(c)
14
MAGNETIC FIELD ALIGNMENT
10c
10c
12c

FIG. 1

FIG.2

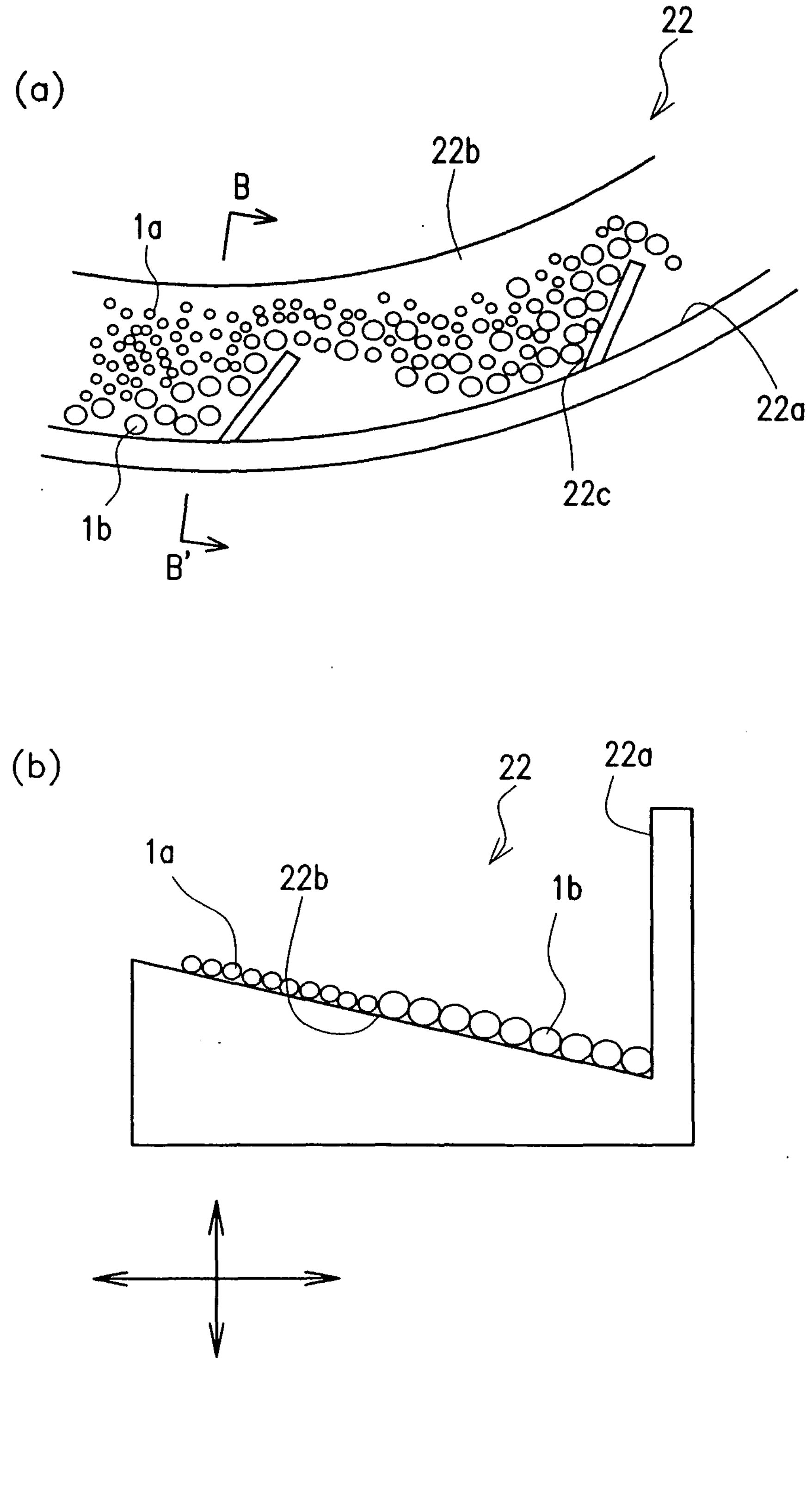


FIG.3

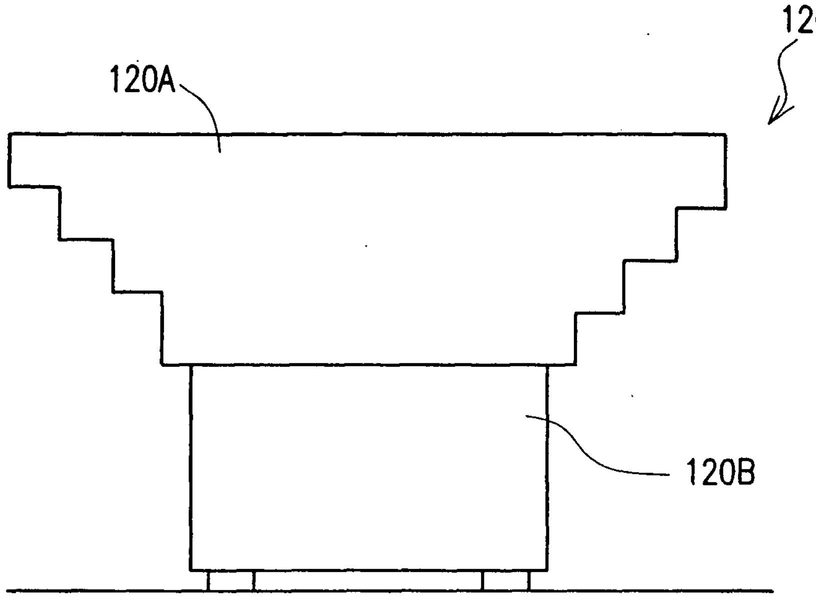


FIG.4

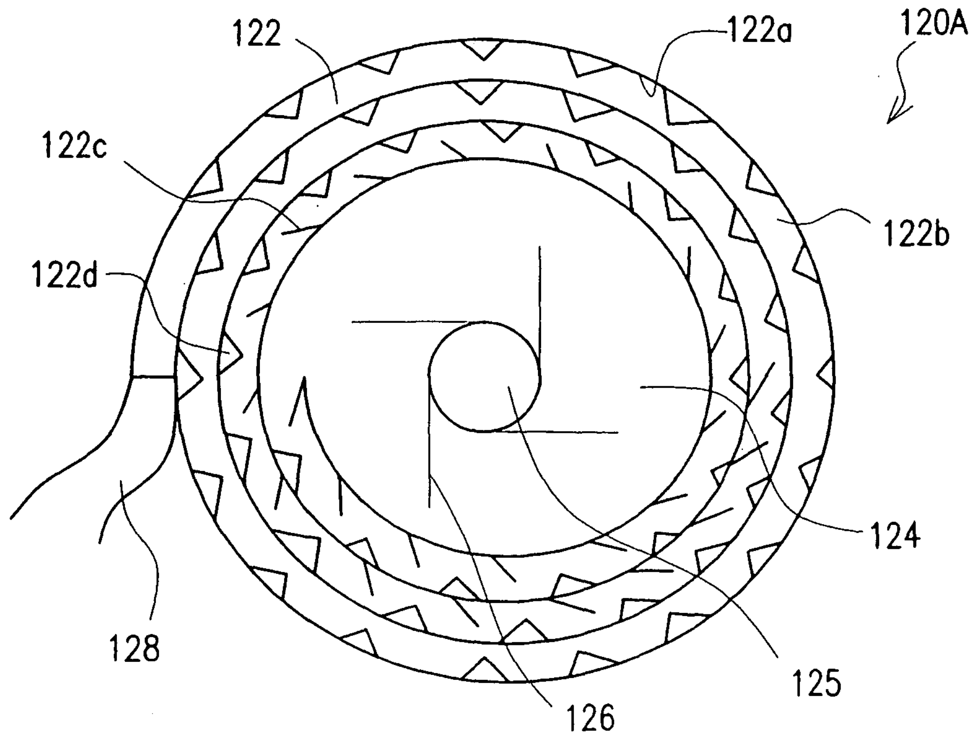


FIG.5

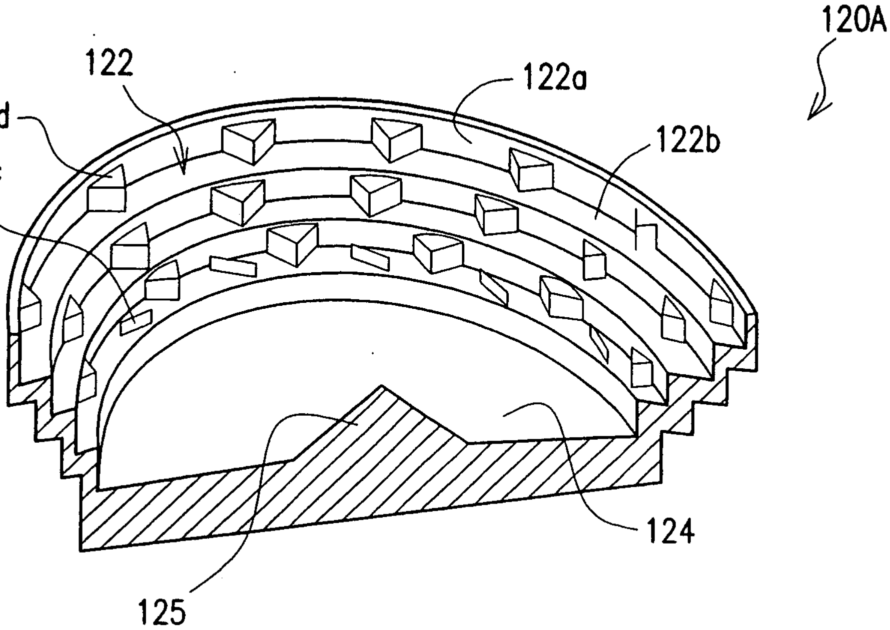
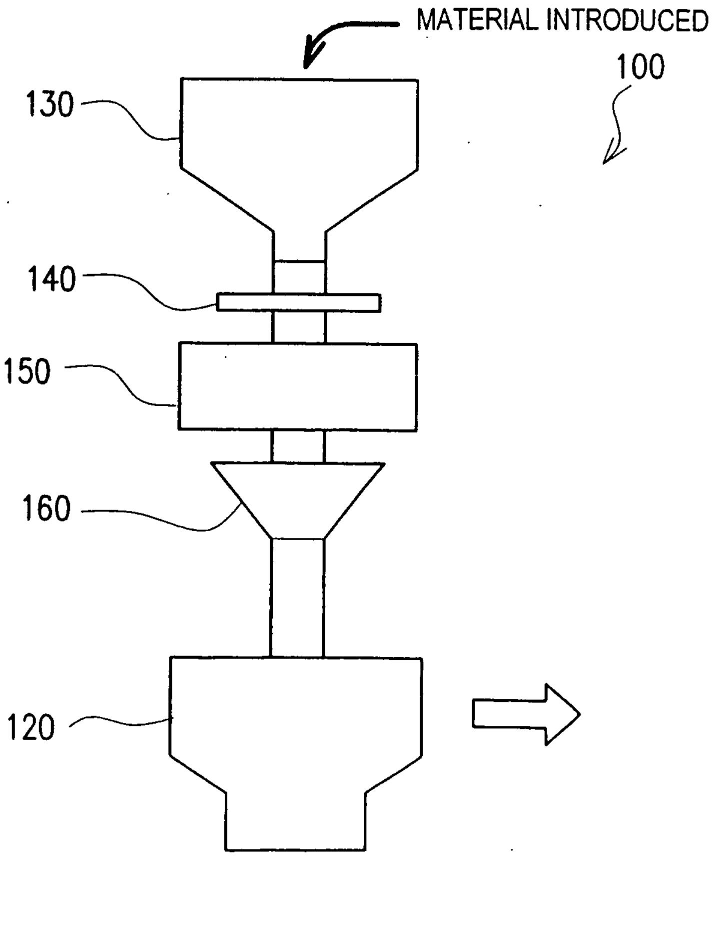

120A
122
122a
122d
122b
122c
124
125

FIG.6


MATERIAL INTRODUCED
100
130
140
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120

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2004/007586

A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl^7 B22F3/02

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)
Int.Cl^7 B22F1/00-3/26, B01J2/18

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

Jitsuyo Shinan Koho	1922-1996	Toroku Jitsuyo Shinan Koho	1994-2004
Kokai Jitsuyo Shinan Koho	1971-2004	Jitsuyo Shinan Toroku Koho	1996-2004

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 01-114005 A (FDK Corp.), 02 May, 1989 (02.05.89), Claims (Family: none)	1-8,10-12
Y	JP 06-262055 A (Daiwa Kasei Kogyo Kabushiki Kaisha), 20 September, 1994 (20.09.94), Claims (Family: none)	1-12
Y	JP 40-022649 B (Shinko Electric Co., Ltd.), 07 October, 1965 (07.10.65), Claims (Family: none)	1-12

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

* Special categories of cited documents:
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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 August, 2004 (10.08.04)	24 August, 2004 (24.08.04)

Name and mailing address of the ISA/	Authorized officer
Japanese Patent Office	
Facsimile No.	Telephone No.

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