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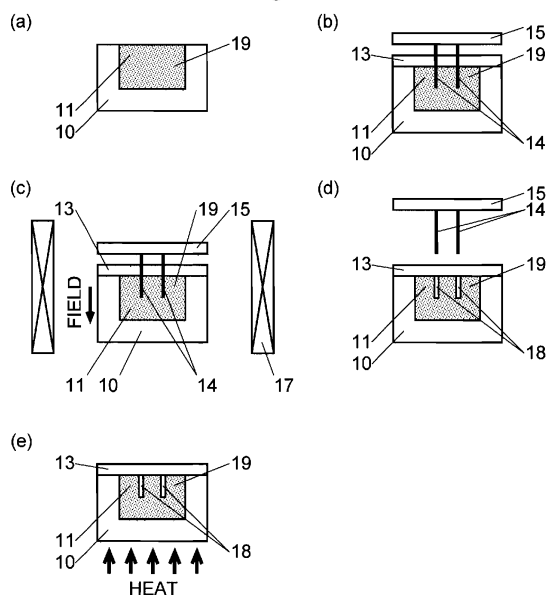
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(54) **METHOD FOR PRODUCING RARE EARTH SINTERED MAGNET AND POWDER CONTAINER FOR RARE EARTH SINTERED MAGNET PRODUCTION**

(57) The present invention is aimed at providing an easy and inexpensive method for producing a sintered rare-earth magnet having cavities, such as slits, for making the magnet less likely to be influenced from eddy currents and/or performing a grain boundary diffusion process. The method for producing a sintered rare-earth magnet according to the present invention is **characterized by** performing the following successive processes: a filling process ((a), (b)) for filling a powder 19 of rare-earth magnet alloy into a powder-filling container 10 together with a cavity-forming member 14; an aligning process (b) for aligning the rare-earth magnet alloy powder 19 in a magnetic field; and a sintering process (e) for sintering the rare-earth magnet alloy powder 19 by heating the rare-earth magnet alloy powder 19 in a state of being held in the powder-filling container 10, wherein (d) the cavity-forming member is removed after the aligning process is completed and before the rare-earth magnet alloy powder begins to be sintered.

Fig.2



Description

TECHNICAL FIELD

5 **[0001]** The present invention relates to a method for producing a sintered rare-earth magnet, such as a sintered Nd-Fe-B magnet or sintered Sm-Co magnet.

BACKGROUND ART

10 **[0002]** Sintered rare-earth magnets are commonly used as permanent magnets capable of creating strong magnetic fields. In particular, sintered Nd-Fe-B magnets are commonly used in motors for hybrid cars or electric vehicles, compact motors for hard-disk drives, large-sized industrial motors, power generators and other applications.

[0003] In such motors or generators, a sintered rare-earth magnet is used as the rotor and an electromagnet as the stator. The electromagnet is operated to create a rotating magnetic field for revolving the rotor. In this process, an eddy current is generated in the sintered rare-earth magnet, causing a loss of energy or overheating of the motor. A technique for solving this problem is disclosed in Patent Document 1, in which a plurality of slits are formed on the surface of the sintered rare-earth magnet to prevent the generation of eddy currents.

[0004] In the case of producing a sintered Nd-Fe-B magnet (neodymium magnet), an alloy powder having a portion of Nd replaced by Dy and/or Tb is used to increase the coercive force of the magnet. However, since Dy and Tb are both expensive and rare elements, this technique increases the production cost and negatively affects the stable supply of the magnet. Another drawback of this technique is the decrease in the maximum energy product. A conventional technique for solving these problems is the grain boundary diffusion, which includes applying Dy and/or Tb to the surface of a sintered compact of Nd-Fe-B alloy containing neither Dy nor Tb and heating it to a temperature within a range from 700 to 1000 degrees Celsius, whereby Dy and/or Tb is transferred through the boundaries of alloy particles into deeper regions of the sintered compact to create a product containing Dy and/or Tb only in the vicinity of the surfaces of the alloy particles. This technique has the effect of achieving a high coercive force while preventing a significant decrease in the maximum energy product as well as decreasing the usage of Dy and Tb. Patent Document 2 discloses the technique of efficiently injecting Dy and/or Tb into the vicinity of the surfaces of alloy particles by forming slits on the surface of the sintered compact of an Nd-Fe-B alloy and diffusing Dy and/or Tb from those slits into the grain boundaries.

BACKGROUND ART DOCUMENT

PATENT DOCUMENT

35 **[0005]**

Patent Document 1: JP-A 2000-295804 (Paragraphs [0009]-[0011])

Patent Document 2: JP-A 2007-053351 (Paragraphs [0027]-[0028] and [0033]-[0035])

DISCLOSURE OF THE INVENTION

PROBLEM TO BE SOLVED BY THE INVENTION

45 **[0006]** In any of the methods described in Patent Documents 1 and 2, the slits are formed by a machining process using a cutter, wire saw or similar tool. The use of a machining process inevitably increases the production cost since it requires a considerable amount of labor and time, with heavy consumption of the tool. Furthermore, the slits created by such a machining process cannot be very thin and hence considerably lower the ratio of the actual volume of the magnet (i.e. the volume of the sintered portion) to its outside volume. As a result, the performance of the product as the magnet substantially deteriorates.

50 **[0007]** In the case where the slits are formed on a compressed compact by machining before sintering, there will be another problem that the alloy powder remaining in the slits cannot be easily removed. If a compact with an alloy powder remaining in the slits is heated for sintering, the alloy powder will partially clog the slits, compromising the effect of preventing the generation of eddy currents. Furthermore, Dy and/or Tb is prevented from sufficiently reaching deep regions in the grain boundary diffusion process.

55 Subjecting a compressed compact to machine work may also cause the additional problems of chipping or cracking.

[0008] Thus, the problem to be solved by the present invention is to provide an easy and inexpensive method for producing a sintered rare-earth magnet having cavities (e.g. slits or holes) for making the magnet less likely to be influenced from eddy currents and/or for performing the grain boundary diffusion process.

MEANS FOR SOLVING THE PROBLEMS

[0009] A method for producing a sintered rare-earth magnet according to the present invention aimed at solving the aforementioned problem is characterized in that a sintered rare-earth magnet having a cavity is produced by performing the following successive processes:

- a) a filling process for filling a powder of rare-earth magnet alloy into a powder-filling container together with a cavity-forming member;
- b) an aligning process for aligning the rare-earth magnet alloy powder in a magnetic field; and
- c) a sintering process for sintering the rare-earth magnet alloy powder by heating the rare-earth magnet alloy powder in a state of being held in the powder-filling container, wherein
- d) the cavity-forming member is removed after the aligning process is completed and before the rare-earth magnet alloy powder begins to be sintered.

[0010] According to the present invention, a sintered rare-earth magnet having a cavity can be easily produced by a simple method including filling a powder of rare-earth magnet alloy into the powder-filling container together with a cavity-forming member and then removing the cavity-forming member before the rare-earth magnet alloy begins to be sintered. Thus, in the present invention, no machining is necessary to create the cavity and a sintered rare-earth magnet having a cavity can be produced at a low cost.

[0011] In most of the conventional methods for producing sintered rare-earth magnets, the compression-molding and aligning of a rare-earth magnet alloy powder is achieved by filling the powder into a container and applying a magnetic field to the powder while compressing it. By contrast, the inventor of the present patent application discovered the fact that a sintered rare-earth magnet could be created by filling a rare-earth magnet alloy powder into a powder-filling container, aligning the rare-earth magnet alloy powder without compression-molding this powder, and heating the powder in a state of being held in the powder-filling container. (This technique is called a press-less method. Refer to JP-A 2006-019521.) In the present invention, since the press-less method is used, the cavity-forming member undergoes no pressure even if this member is put in the powder-filling container together with the rare-earth magnet alloy powder.

[0012] As a result of the aligning process in the magnetic field, the particles of the rare-earth magnet alloy powder held in the powder-filling container magnetically attract each other. In the present invention, since the cavity-forming member is removed after the aligning process, the cavity will not be destroyed when the cavity-forming member is removed.

[0013] When the rare-earth magnet alloy powder is heated to higher temperatures in the sintering process, the powder begins to be sintered when its temperature exceeds a specific level (e.g. approximately 600 degrees Celsius for a sintered Nd-Fe-B magnet), after which the sintered compact shrinks as the sintering process continues. To avoid impeding this shrinkage, the cavity-forming member used in the present invention is removed before the rare-earth magnet alloy powder begins to be sintered.

[0014] The removal of the cavity-forming member may be performed before the sintering process is initiated. This is desirable in that it eliminates the necessity of considering the heat resistance of the cavity-forming member or the reactivity between the cavity-forming member and the rare-earth magnet alloy powder.

[0015] It is possible to use a cavity-forming member that liquefies or vaporizes at a temperature lower than the temperature at which the sintering begins. In this case, the cavity-forming member will be removed after the temperature begins to increase for the sintering and before the sintering actually begins.

[0016] If the aforementioned rare-earth magnet alloy is an alloy of a sintered Nd-Fe-B magnet, Dy and/or Tb can be diffused into the sintered compact by injecting a substance containing Dy and/or Tb into the cavity of the sintered compact obtained by the sintering process.

[0017] If slits for preventing the influence of eddy currents need to be formed on the sintered rare-earth magnet, a plate-shaped member can be used as the cavity-forming member. If the grain boundary diffusion is of primary importance, a rod-shaped member may be used. In the latter case, a large number of rod-shaped cavity-forming members may be arranged in the form of a matrix, whereby Dy and/or Tb can be uniformly diffused from a large number of holes. The cross-sectional shape of the rod-shaped cavity-forming member is not specifically limited; for example, it may be circular, quadrilateral or hexagonal.

[0018] If a plate-shaped or rod-shaped cavity-forming member is used as the cavity-forming member, it is preferable to align the rare-earth magnet alloy powder in a magnetic field parallel to the cavity-forming member in the aligning process. The particles of the rare-earth magnet alloy powder forms a chain-like structure extending in the direction parallel to the cavity-forming member. Therefore, even if the cavity-forming member is removed in this state, the chain-like structure will not be broken off and the cavity will remain undestroyed.

[0019] To assuredly prevent the destruction of the cavity, the rare-earth magnet alloy powder may be mixed with a binder when it is filled into the powder-filling container. Examples of the binder include methyl cellulose, polyacrylamide,

polyvinyl alcohol, paraffin wax, polyethylene glycol, polyvinyl pyrrolidone, hydroxypropyl cellulose, hydroxypropyl methylcellulose, ethyl cellulose, acetyl cellulose, nitrocellulose, and polyvinyl acetate resin. (Refer to JP-A 10-270278.)

[0020] When the rare-earth magnet alloy powder is filled into the powder-filling container together with the cavity-forming member, it is possible to simultaneously put both the powder of rare-earth magnet alloy and the cavity-forming member into the powder-filling container or to separately and sequentially fill them into the container.

[0021] The cavity formed in the sintered compact by the production method according to the present invention is mechanically weak and rather fragile if left in its original state. Furthermore, the cavity may retain moisture and cause corrosion or mechanical destruction of the product. To avoid these problems, an embedding member, such as epoxy resin, may be filled into the cavity to increase its mechanical strength and prevent the retention of moisture. The process of filling the embedding member is performed after the removal of the cavity-forming member. If the embedding member is an epoxy resin or similar material whose heat-resistant temperature is lower than the sintering temperature of the rare-earth magnet, the filling process is performed after the sintering process. If the diffusion process is additionally performed, the filling process is performed after the diffusion process. The embedding member should desirably be made of an insulating material to prevent the influence of eddy currents.

EFFECT OF THE INVENTION

[0022] With the present invention, a cavity can be formed by a simple method including filling a powder of rare-earth magnet alloy into a powder-filling container together with a cavity-forming member, aligning the powder in a magnetic field, and removing the cavity-forming member. By this method, a sintered rare-earth magnet having a cavity can be easily produced at a low cost since no machining is required.

BRIEF DESCRIPTION OF THE DRAWINGS

[0023]

Fig. 1 shows vertical-sectional views of the first embodiment of the mold, a lid for this mold, and a cavity-forming member used in a method for producing a sintered rare-earth magnet according to the present invention, as well as a top view of the aforementioned lid.

Fig. 2 is a schematic diagram showing the first embodiment of the method for producing a sintered rare-earth magnet according to the present invention.

Fig. 3 shows vertical-sectional views of the second embodiment of the mold, a lid for this mold, and a cavity-forming member used in a method for producing a sintered rare-earth magnet according to the present invention, as well as a bottom view of the aforementioned mold.

Fig. 4 is a schematic diagram showing the second embodiment of the method for producing a sintered rare-earth magnet according to the present invention.

Fig. 5 shows vertical-sectional views of another example of the mold and a lid for this mold in the present invention.

Fig. 6 is a perspective view showing one embodiment of the rod-shaped cavity-forming member.

Fig. 7 is a schematic diagram showing one embodiment of the grain boundary diffusion process in the present invention.

Fig. 8 is a schematic diagram showing one embodiment of the process for filling an embedding member into the cavities.

Fig. 9 is a perspective view of a sintered rare-earth magnet created by the method according to Embodiment 1.

Fig. 10 shows vertical-sectional views of the mold, a lid for this mold, and a cavity-forming member used in Example 3-1, as well as a top view of the aforementioned lid.

Fig. 11 shows vertical-sectional views of the mold, a lid for this mold, and a cavity-forming member used in Example 3-2, as well as a top view of the aforementioned lid.

BEST MODES FOR CARRYING OUT THE INVENTION

[0024] Embodiments of the method for producing a sintered rare-earth magnet according to the present invention are hereinafter described by means of Figs. 1-11.

Figs. 1 and 2 show the first embodiment of the present invention. The method according to the first embodiment uses a mold (powder-filling container) 10 and a cavity-forming member 14 shown in Fig. 1. The mold 10, which is designed for creating a plate-shaped magnet, has a rectangular-parallelepiped receiving section 11, into which a powder of rare-earth magnet alloy is to be filled. This receiving section 11 has an opening on its upper side, thus allowing the filling of the rare-earth magnet alloy powder and removal of a sintered rare-earth magnet after the sintering process. A lid 13 for closing this opening is attached thereto. Examples of the materials available for the mold 10 and the lid 13 include

magnetic stainless steel, non-magnetic stainless steel, and some types of carbon that are heat-resistant to temperatures equal to or higher than the sintering temperature used for creating the sintered rare-earth magnet. The lid 13 has two insertion openings 131 extending parallel to each other in the longitudinal direction of the rectangular-parallelepiped receiving section 11. Each insertion opening 131 allows the insertion of a plate-shaped cavity-forming member 14, which is slightly smaller than the insertion opening 131 in both width and length. Examples of the materials available for the cavity-forming member 14 include various kinds of metal, carbon and plastic (which do not need to be heat-resistant in the present embodiment). There are two cavity-forming members 14 standing on a plate-shaped attachment base 15, with the same interval as the two insertion openings 131.

[0025] The method for producing a sintered rare-earth magnet according to the present embodiment is hereinafter described by means of Fig. 2. Initially, a rare-earth magnet alloy powder 19 is filled in the receiving section 11 (Fig. 2(a)). In this step, the rare-earth magnet alloy powder 19 in a pure form may be used, or a binder may be mixed with the rare-earth magnet alloy powder 19. The filling density should preferably be within a range from 40 to 50 % of the true density of the rare-earth magnet alloy powder. Next, the lid 13 is attached to the mold 10, and the cavity-forming members 14 are inserted through the insertion openings 131 into the rare-earth magnet alloy powder 19 held in the receiving section 11 (Fig. 2(b)). Subsequently, the mold 10 is set into a magnetic-field generation coil 17, and a pulsed magnetic field parallel to the cavity-forming members 14 (and perpendicular to the lid 13) is applied to align the rare-earth magnet alloy powder 19 (Fig. 2(c)). The strength of this magnetic field should be within a range from 3 to 10 T, and more preferably from 4 to 8 T. While the magnetic field is applied, the lid 13 should be securely pressed onto the mold 10 to prevent the rare-earth magnet alloy powder 19 from escaping. After the aligning process in the magnetic field, the cavity-forming members 14 are pulled out from the rare-earth magnet alloy powder 19 and the insertion openings 131 (Fig. 2(d)). Thus, slit-shaped cavities 18 are formed in the compact of the rare-earth magnet alloy powder 19. As a result of the aligning process in the magnetic field, the fine particles of the powder magnetically attract each other and hence will barely fall into the cavities 18. Subsequently, the rare-earth magnet alloy powder 19 in a state of being held in the receiving section 11 is heated (Fig. 2(e)). Thus, a sintered rare-earth magnet having slit-shaped cavities is obtained. During the sintering process, water and other substances that are inevitably present in the rare-earth magnet alloy powder 19 vaporize, and the generated gas is discharged through the insertion openings 131 to the outside of the mold.

[0026] By this method, the slits can be created at a much lower cost than in the case of performing machine work using a wire saw or similar tool after the sintering process. Furthermore, a narrow slit that cannot be created by machining can be created. The obtained slits are completely free from any unwanted matter (e.g. residual powder inside the slits) which lowers the functionalities of the slits. Thus, a high-quality slit can be obtained.

[0027] Figs. 3 and 4 show the second embodiment of the present invention. The method according to the second embodiment uses a mold 20 shown in Fig. 3 and a cavity-forming member 24 shown in Fig. 4. Similar to the mold 10 used in the first embodiment, the mold 20 has a receiving section 21 to which a lid 23 can be attached. A difference from the first example exists in that two insertion openings 221 are formed in the bottom of the mold 20. No insertion opening is formed in the lid 23. Similar to the first embodiment, the cavity-forming members 24 fixed to a cavity-forming member attachment base 25 can be inserted into the insertion openings 221.

[0028] The method for producing a sintered rare-earth magnet according to the second embodiment is hereinafter described by means of Fig. 4. Initially, the cavity-forming members 24 are inserted into the insertion openings 221 of the mold 20 (Fig. 4(a)). Next, a rare-earth magnet alloy powder 29 is filled in the receiving section 21, and the lid 23 is attached (Fig. 4(b)). Thus, the insertion of the cavity-forming member and the filling of the rare-earth magnet alloy powder are performed in reverse order as compared to the first embodiment. Next, the mold 20 is set into a magnetic-field generation coil 27, and a pulsed magnetic field parallel to the cavity-forming members 24 (and perpendicular to the lid 23) is applied to align the rare-earth magnet alloy powder 29 (Fig. 4(c)). Subsequently, the cavity-forming members 24 are pulled out from the rare-earth magnet alloy powder 29 and the insertion openings 221 to form cavities 28 (Fig. 4(d)), and the rare-earth magnet alloy powder 29 in a state of being held in the receiving section 21 is sintered by heat (Fig. 4(e)).

[0029] Fig. 5 shows another example of the mold. Unlike the mold 10 shown in Fig. 1 in which the cavity-forming members 14 are fixed to the cavity-forming member attachment base 15 prepared separately from the lid 13, the cavity-forming members 14A in the present example are directly fixed to the lid 13A (Fig. 5(a)). If this lid 13A is used, the lid 13A is detached from the mold after the aligning process in order to remove the cavity-forming members 14.

[0030] The previous descriptions pertained to the cases where the cavity-forming members are removed after the aligning process. On the other hand, if the cavity-forming members are made of a material that liquefies or vaporizes at a temperature lower than the sintering temperature of the rare-earth magnet alloy powder, it is possible to remove the cavity-forming members, without pulling them out, by heating them together with the mold and rare-earth magnet alloy powder. In this case, the cavity-forming members may be attached to the inside of the receiving section. Specific examples of the materials available for such a cavity-forming member include polyvinyl alcohol or other plastic materials that easily vaporize. Fig. 5(b) shows one example in which cavity-forming members 14B stand at the bottom 12 of the receiving section 11.

[0031] The following description explains how to determine an appropriate thickness and interval of the cavity-forming

members as well as an appropriate depth by which these members should be inserted into the rare-earth magnet alloy powder (which is hereinafter called the "insertion depth").

Initially, an appropriate width, insertion depth, number and interval of the cavity-forming members will be explained for the case where the primary purpose of the cavities is to prevent eddy current during the usage of the sintered rare-earth magnet. In this case, the intended objective, i.e. the prevention of the eddy currents, can be achieved even if the slit is too narrow. Therefore, in order to improve the inherent performance of the magnet, the slits formed in the sintered compact should be as narrow as possible. This means that the cavity-forming members should be as thin as possible. For example, in the case of using a member similar to a razor blade, which is a typical example of the thin plate-shaped member, the lower limit of the thickness of the cavity-forming member is approximately 0.05 mm. In this case, with the sintering shrinkage taken into account, the width of the slit to be eventually formed in the sintered compact will be approximately 0.04 mm. With respect to the insertion depth, it is preferable to increase this depth to improve the effect of reducing the eddy currents. However, to ensure an adequate mechanical strength of the sintered compact, the depth should be smaller than the magnet's thickness in the direction of the insertion depth by 1 mm or more, and more preferably 2 mm or more.

If the cavity-forming member is excessively thick, the volume ratio of the magnet (i.e. the ratio of the volume where the magnet actually exists to the outside volume of the sintered magnet) will be too low and the magnetic properties of the product will deteriorate. Accordingly, the thickness of the cavity-forming member should be appropriately determined so that the volume ratio will be equal to or higher than 90 %.

With respect to the interval of the slits, or the interval of the cavity-forming members, it is preferable to reduce this interval since the loss of energy due to the eddy currents generated in the magnet is proportional to the second power of the magnet size. However, increasing the number of slits reduces the volume ratio of the magnet. Given these factors along with the aforementioned conditions relating to the thickness and insertion depth, the interval and number of the cavity-forming members should be determined so that the volume ratio will exceed the level where the required magnetic properties are obtained.

[0032] Next, an appropriate width, insertion depth, number and interval of the cavity-forming members will be explained for the case where the primary purpose of the cavities is to help the grain boundary diffusion of Dy and/or Tb into the sintered compact. If the cavity-forming member is too narrow, it is difficult to inject a substance containing Dy and/or Tb into the slit formed in the sintered compact. Therefore, it is preferable to form the slits with a width equal to or larger than 0.1 mm. If the interval of the slits is too large, the effect of grain boundary diffusion cannot extend over the entirety of the sintered magnet, causing the resulting product to have uneven magnetic properties. Accordingly, the interval of the slits, or the interval of the cavity-forming members, should preferably be equal to or smaller than 6 mm, and more preferably equal to or smaller than 5 mm. With respect to the insertion depth, the difference between this depth and the magnet's thickness in the direction of the insertion depth should preferably be equal to or smaller than 6 mm, and more preferably equal to or smaller than 5 mm. However, to ensure an adequate mechanical strength of the sintered compact, the difference should preferably be equal to or larger than 1 mm, and more preferably equal to or larger than 2 mm. Additionally, as in the previous case where the primary purpose was to prevent the eddy current, the thickness, insertion depth, number and interval of the cavity-forming members should be determined so that the volume ratio of the product will exceed the level where the required magnetic properties are obtained.

[0033] The previous examples illustrated the case of using a plate-shaped cavity-forming member. If the primary purpose is to help the grain boundary diffusion, it is possible to use a rod-shaped cavity-forming member. Fig. 6 shows one example, in which a large number of rod-shaped cavity-forming members 34 are arrayed in rows and columns in the form of a matrix on a plate-shaped attachment base 35. The use of such a large number of rod-shaped cavity-forming members 34 in the form of a matrix results in a sintered compact having a large number of fine pores (cavities). When a grain boundary diffusion process is performed to create a sintered Nd-Fe-B magnet, Dy and/or Tb can be efficiently diffused through these fine pores into the sintered compact.

[0034] To ensure the injection of a substance containing Dy and/or Tb, the diameter of the fine pores formed in the sintered compact should preferably be equal to or larger than 0.2 mm, and more preferably equal to or larger than 0.3 mm. The interval of the cavity-forming members 34 should preferably be equal to or smaller than 6 mm, and more preferably equal to or smaller than 6 mm, to diffuse Dy and/or Tb over the entirety of the sintered magnet. The conditions to be considered for the insertion depth are the same as in the case of the plate-shaped cavity-forming member.

[0035] The diffusion process includes filling a powder containing Dy and/or Tb into the cavities 18 and then heating the filled powder (Fig. 7). The heating temperature is typically within a range from 700 to 1000 degrees Celsius. The Dy/Tb-containing substance to be injected into the cavities may be a fluoride, oxide, acid fluoride or hydride of Dy or Tb, an alloy of Dy or Tb and another kind of metal, or a hydride of such an alloy. Examples of the alloy of Dy or Tb and another kind of metal include alloys of Ty or Tb and an iron group transition metal (e.g. Fe, Co or Ni), B, Al or Cu. The grain boundary diffusion process can be effectively performed by mixing the aforementioned substances in an organic or similar solvent to prepare a slurry, injecting this slurry into the cavities, and heating the slurry. This slurry may be injected into the cavities only, or it may be additionally applied to the surface of the sintered compact. In latter case, the

grain boundary diffusion takes place from both the cavities and the surface of the sintered compact. After the slurry is injected into the cavities of the sintered compact (and applied to its surface in some cases), the grain boundary diffusion process is performed by heating the sintered compact at 700 to 1000 degrees Celsius for one to twenty hours under vacuum or in an inert-gas atmosphere. This grain boundary diffusion process uses only a small amount of Dy and/or Tb and yet can effectively increase the coercive force of the sintered Nd-Fe-B magnet without significantly decreasing its residual flux density even if the magnet has a substantially large thickness of 5 mm or larger.

[0036] In the case where the cavities are formed for both purposes of helping the grain boundary diffusion process and reducing the loss of energy due to eddy currents, if the aforementioned slurry is used in the grain boundary diffusion process, it is necessary to control the amount of the slurry so that an electrically conductive component of the injected slurry will not fill the cavity.

[0037] In any of the previously described embodiments, it is possible to fill the cavities with an epoxy resin or similar embedding member to prevent a decrease in the mechanical strength of the product due to the presence of the cavities and the corrosion or other problems due to retention of moisture in the cavities. In this case, an epoxy resin in a liquid state is injected into the cavities 18 and then cured at room temperature or by heat (Fig. 8). For some type of material of the embedding member, this embedding process can be performed before the sintering process. In the case of using an epoxy resin or similar adhesive resin, this process is performed after the sintering process. If the diffusion process is additionally performed, the embedding process is performed after the diffusion process.

EXAMPLE 1

[0038] A strip-cast alloy of an Nd-Fe-B rare-earth magnet was subjected to hydrogen pulverization and then a jet-mill process using nitrogen gas, to obtain a rare-earth magnet powder with an average particle size of 5 μm . The composition of this rare-earth magnet powder ratio was Nd: 25.8%, Pr: 4.3%, Dy: 2.5%, Al: 0.23%, Cu: 0.1%, and Fe: the rest. The average particle size of the rare-earth magnet powder was measured with a laser-type particle-size analyzer.

[0039] This powder was filled into the mold 10 of the first embodiment to an apparent density of 3.5 g/cm³, after which the lid 13 was put on the mold 10. Subsequently, the cavity-forming members 14 were inserted through the insertion openings 131. After the mold 10 was fixed in a magnetic-field generation coil, a pulsed magnetic field of 5 T was applied three times in the direction parallel to the cavity-forming members 14 and perpendicular to the bottom of the mold 10 so as to align the rare-earth magnet powder in the magnetic field. Subsequently, the cavity-forming members 14 were pulled out from the mold 10, and then the mold 10 was put into a sintering furnace. The entire process from the filling of the powder to the putting of the mold into the furnace was carried out in an argon-gas atmosphere. The sintering process was performed under vacuum at 1010 degrees Celsius for two hours. In this example, the mold 10 and the lid 13 were made of carbon and the cavity-forming members 14 were made of non-magnetic stainless steel. The thickness of the cavity-forming members 14 was 0.5 mm.

[0040] The sintered compact created by the previously described process had a density of 7.56 g/cm³, which is as high as the density of a sintered Nd-Fe-B magnet created by a normal pressing method. The obtained sintered compact 31 (Fig. 9) had the shape of a rectangular parallelepiped having a short-side length of 37 mm, a long-side length of 39 mm and a height of 8.6 mm, with two slits 32 extending parallel to the shorter sides and at an interval of 12 mm on the top face. No noticeable deformation in the outside shape of the sintered compact or the slits 32 was recognized. The slits 32 had a width of approximately 0.4 mm and a depth of 6.2 mm. For inspection, a metallic foil having a thickness of 0.3 mm was inserted into each slit 32. The result confirmed that none of these slits 32 was clogged or closed with foreign matter.

EXAMPLE 2

[0041] Using the same powder as used in Example 1, a sintered Nd-Fe-B magnet with slits was created by using the mold 20 and the cavity-forming members 24 of the second embodiment. When the mold 20 of the second embodiment is used, the powder needs to be filled into the mold 20 with the cavity-forming members 24 attached thereto. In filling the powder, it is necessary to carefully fill it so that the powder will be uniformly put into the entire receiving section 21. The filling density was 3.6 g/cm³. After the powder was filled, the lid 23 was put on the mold 20. Subsequently, the aligning process in the magnetic field and the removal of the cavity-forming members 24 were performed under the same conditions as in Example 1, and then the sintering process was performed under the same conditions as in Example 1. After the sintering process, the sintered compact was removed from the mold. Similar to the product created in Example 1, the obtained sintered compact had a high density and no deformation in its shape. The slits were also found to be high-quality slits free from clogging or closing. The outside shape of the sintered compact, the interval of the slits, the width and other sizes of each slit were approximately the same as those of Example 1.

EXAMPLE 3

[0042] A sintered compact with cavities (slits or fine pores) was created by using the molds and cavity-forming members shown in Figs. 10 and 11. The mold 40 shown in Fig. 10 has a rectangular-parallelepiped receiving section 41 having square-shaped top and bottom sides. A lid 43 can be attached to the top side. This lid 43 has two insertion openings 431 for allowing the insertion of two plate-shaped cavity-forming members 44. The mold used in the example shown in Fig. 11 is the same as this mold 40. A lid 53 to be attached to the mold 40 in the latter example has four insertion openings 531 arranged in the form of a square, thus allowing the insertion of four rod-shaped cavity-forming members 54.

[0043] Using the same rare-earth magnet powder and method as used in Example 1, a sintered compact with slits (Example 3-1) and a sintered compact with fine pores (Example 3-2) were created by using the cavity-forming members 44 and 54, respectively. Both sintered compacts had a cubic outside shape with one side approximately measuring 11 mm. The slits formed in the former sintered compact had a width of 0.4 mm and a depth of 5.9 mm, and were spaced by an interval of 3.3 mm. The fine pores formed in the latter sintered compact had a diameter of 0.5 mm and a depth of 7.2 mm. For comparison, another sintered compact having a rectangular-parallelepiped shape with neither slits nor fine pores (Comparative Example 1) was also created under the same conditions as used in the present Example (and Example 1) except that the insertion and removal of the cavity-forming members 44 were omitted. Each of the three types of sintered compacts was shaped into a cube with one side accurately measuring 10 mm by using a surface grinder. The obtained cubes were then subjected to alkaline cleaning, acid cleaning and pure-water cleaning processes followed by a drying process.

[0044] For these samples, a grain boundary diffusion process using a Dy-containing alloy powder was performed as follows: Initially, a Dy-containing alloy having a composition by atomic ratio of Dy: 80%, Ni: 14%, Al: 4%, and other kinds metals and impurities: 2 % was pulverized to an average particle size of 9 μm with a jet mill to obtain a Dy-containing alloy powder. Next, this powder was mixed with ethanol by 50 % by weight and stirred. The obtained mixture was vacuum-impregnated into the slits of the sample of Example 3-1 and the fine pores of the sample of Example 3-2, and then dried. Subsequently, the Dy-containing powder was applied to the surface of each of the magnets of Examples 3-1, 3-2 and Comparative Example. These three types of sintered compacts were put into a vacuum furnace and heated at 900 degrees Celsius for three hours. After that, they were rapidly cooled to room temperature, then heated to 500 degrees Celsius, and again rapidly cooled to room temperature. The magnetic properties of the three samples created in this manner are shown in Table 1. In this table, Comparative Example 1-1 was obtained by performing the aforementioned grain boundary diffusion process on the sintered compact of Comparative Example 1. Comparative Example 1-2 was obtained by heating the sintered compact of Comparative Example 1, without any Dy-containing alloy powder applied to its surface, in the same manner as in the grain boundary diffusion process.

[0045]

TABLE 1

	Residual Flux Density Br[kG]	Coercive Force H_{cJ} [kOe]	Maximum Energy Product $(BH)_{\text{max}}$ [MGOe]	Ratio of Br to Saturated Magnetization Br/J _s [%]	Squareness H_k/H_{cJ} [%]
Example 3-1	12.7	28.6	39.8	94.3	93.7
Example 3-2	12.8	28.2	40.0	94.5	95.2
Comparative Example 1-1	13.0	24.6	41.2	94.3	72.2
Comparative Example 1-2	13.0	21.6	41.4	94.4	94.3

[0046] As compared to the sample of Comparative Example 1-1, for which the grain boundary diffusion process was performed with neither slits nor fine pores, the samples of Examples 3-1 and 3-2 had higher coercive forces H_{cJ} and higher squareness H_k/H_{cJ} of magnetization curves. Their coercive forces H_{cJ} were also higher than that of the sample of Comparative Example 1-2, for which no grain boundary diffusion process was performed. These results demonstrate that the method according to the present invention, which is an inexpensive method that does not include the expensive machining process for forming slits after the sintering process, is effective for enhancing the coercive force of a sintered Nd-Fe-B magnet by grain boundary diffusion even in the case where the magnet is large sized, like the 10-mm cube, for which the grain boundary diffusion process has not been effective before.

EXPLANATION OF NUMERALS

[0047]

5	10, 20, 40	Mold (Powder-Filling Container)
	11, 21, 41	Receiving Section of Mold
	12	Bottom of Mold
10	13, 23, 53	Lid of Mold
	131, 221, 431, 531	Insertion Opening
15	14, 24, 34, 44, 54	Cavity-Forming Member
	15, 25, 35	Attachment Base for Cavity-Forming Members
	17, 27	Magnetic-Field Generation Coil
20	18, 28	Cavity
	19, 29	Rare-Earth Magnet Alloy Powder
25	31	Sintered Compact

Claims

- 30 1. A method for producing a sintered rare-earth magnet, **characterized in that** a sintered rare-earth magnet having a cavity is produced by performing following successive processes:
- a) a filling process for filling a powder of rare-earth magnet alloy into a powder-filling container together with a cavity-forming member;
- 35 b) an aligning process for aligning the rare-earth magnet alloy powder in a magnetic field; and
- c) a sintering process for sintering the rare-earth magnet alloy powder by heating the rare-earth magnet alloy powder in a state of being held in the powder-filling container, wherein
- d) the cavity-forming member is removed after the aligning process is completed and before the rare-earth magnet alloy powder begins to be sintered.
- 40 2. The method for producing a sintered rare-earth magnet according to claim 1, **characterized in that** the removal of the cavity-forming member is performed before the sintering process is initiated.
3. The method for producing a sintered rare-earth magnet according to claim 1 or 2, **characterized in that** the cavity-forming member is a plate-shaped member or a rod-shaped member.
- 45 4. The method for producing a sintered rare-earth magnet according to claim 3, **characterized in that** the rare-earth magnet alloy powder is aligned in a magnetic field parallel to the cavity-forming member in the aligning process.
- 50 5. The method for producing a sintered rare-earth magnet according to one of claims 1-4, **characterized in that** a binder is filled into the powder-filling container together with the rare-earth magnet alloy powder in the filling process.
6. The method for producing a sintered rare-earth magnet according to one of claims 1-5, **characterized in that** an embedding member is filled into the cavity after the removal of the cavity-forming member.
- 55 7. The method for producing a sintered rare-earth magnet according to one of claims 1-5, **characterized in that**:
- the rare-earth magnet alloy is an Nd-Fe-B magnet alloy; and

a diffusing process for diffusing Dy and/or Tb into the sintered compact is performed by injecting a substance containing Dy and/or Tb into the cavity of the sintered compact obtained by the sintering process.

5 8. The method for producing a sintered rare-earth magnet according to claim 7, **characterized in that** an embedding member is filled into the cavity after the diffusing process.

9. The method for producing a sintered rare-earth magnet according to claim 6 or 8, **characterized in that** the embedding member is made of an insulating material.

10 10. The method for producing a sintered rare-earth magnet according to one of claims 1-9, **characterized in that** the filling process includes inserting the cavity-forming member into the powder-filling container through an insertion opening formed in either the powder-filling container or a lid of the powder-filling container, and the removal process includes pulling out the cavity-forming member from the insertion opening.

15 11. A powder-filling container for producing a sintered rare-earth magnet, **characterized by** comprising:

 a mold into which a powder of rare-earth magnet alloy is to be filled;
 a lid to be attached to the mold; and
 an insertion opening, formed in either the mold or the lid, for allowing insertion of a cavity-forming member.

20 12. A powder-filling container for producing a sintered rare-earth magnet, **characterized by** comprising:

 a mold into which a powder of rare-earth magnet alloy is to be filled;
 a lid to be attached to the mold; and
25 a cavity-forming member provided on either the mold or the lid.

Fig. 1

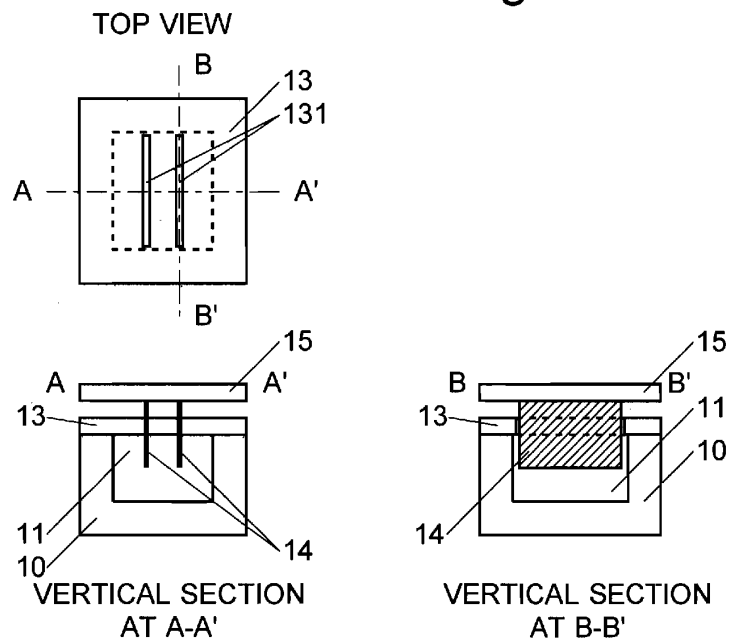


Fig.2

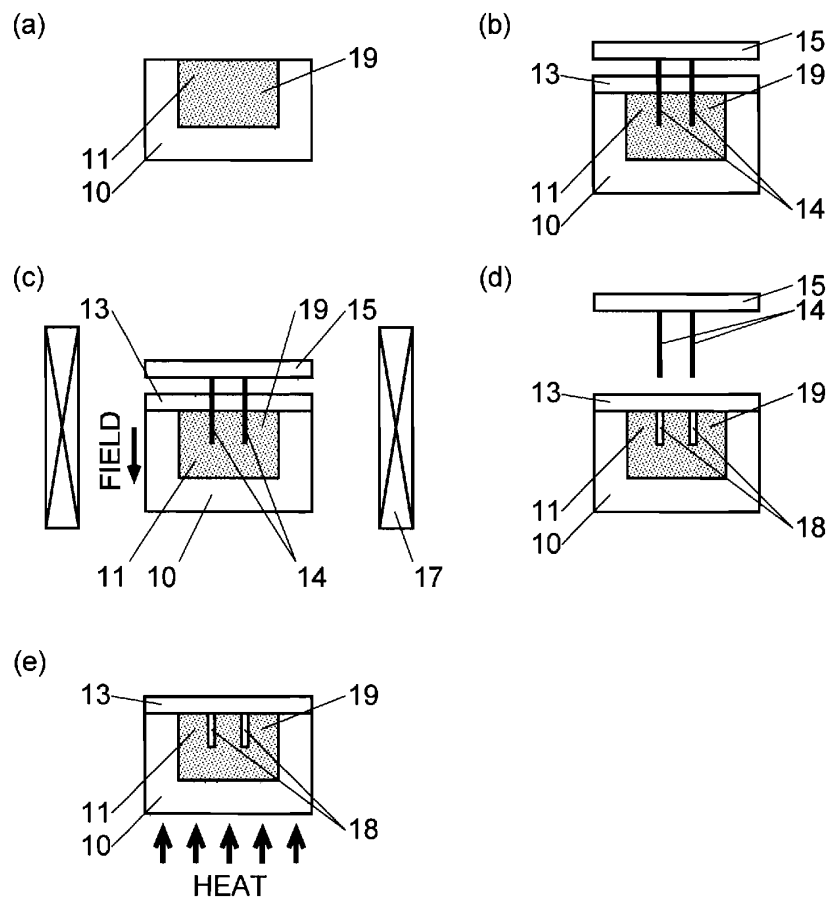


Fig. 3

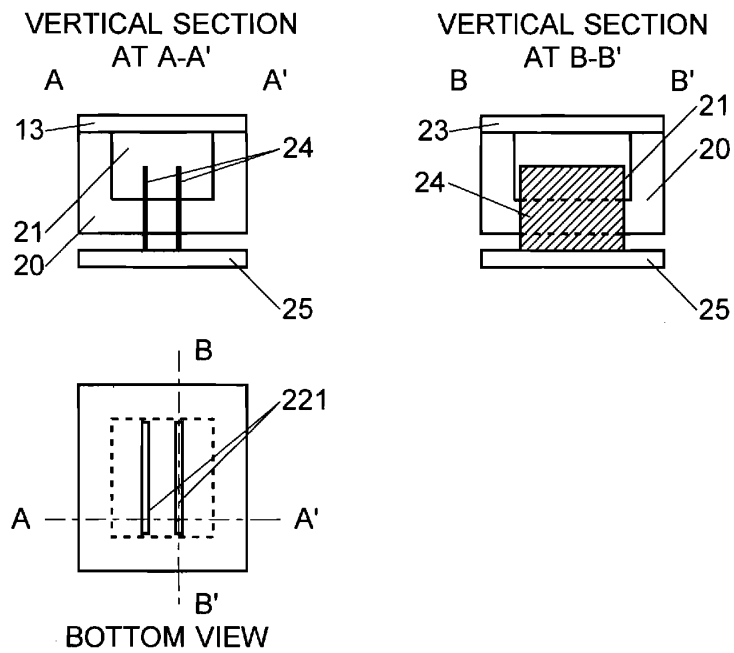


Fig. 4

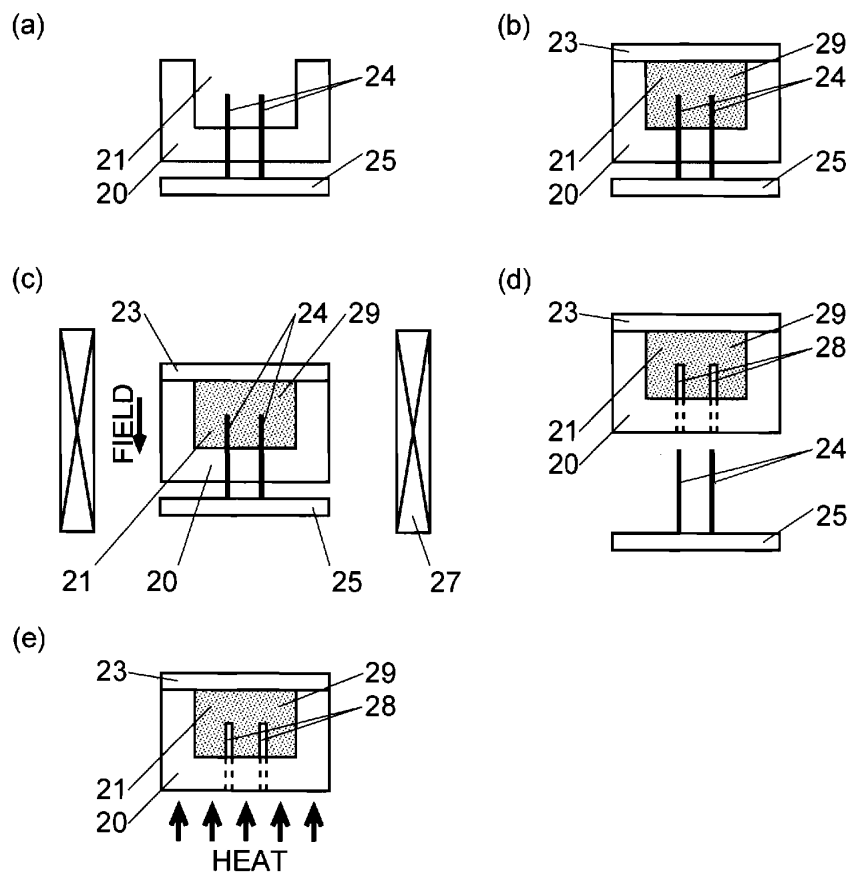


Fig. 5

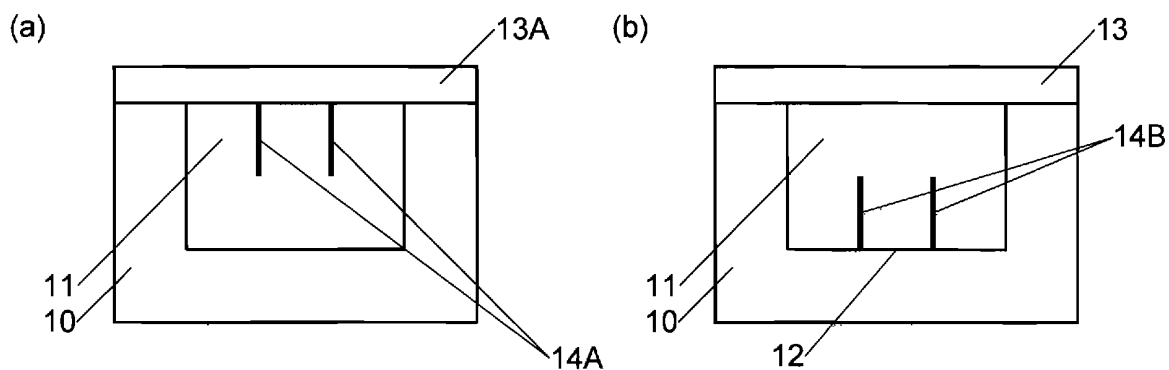


Fig. 6

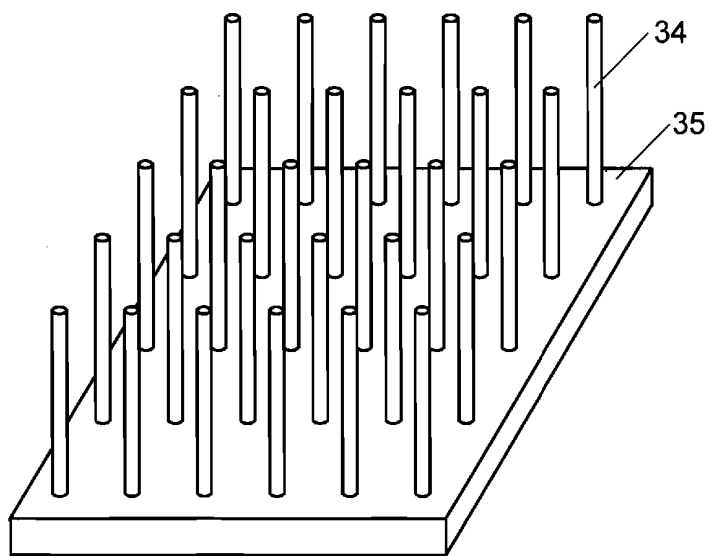


Fig.7

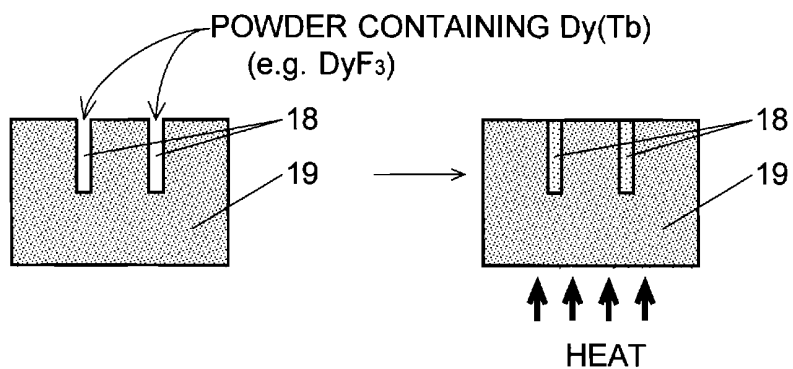


Fig. 8

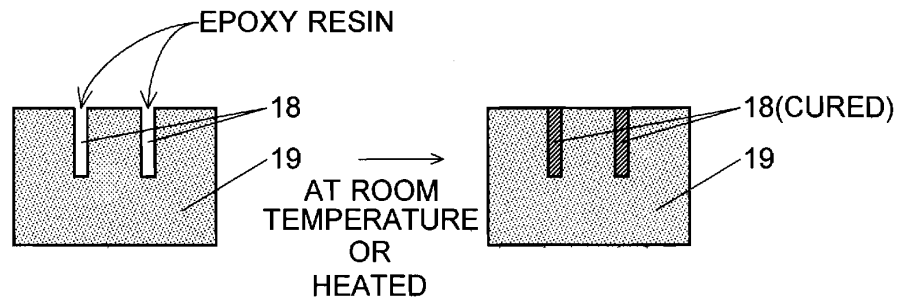


Fig. 9

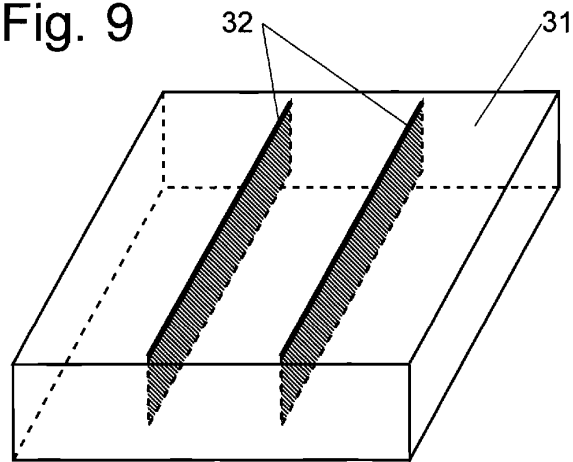


Fig. 10

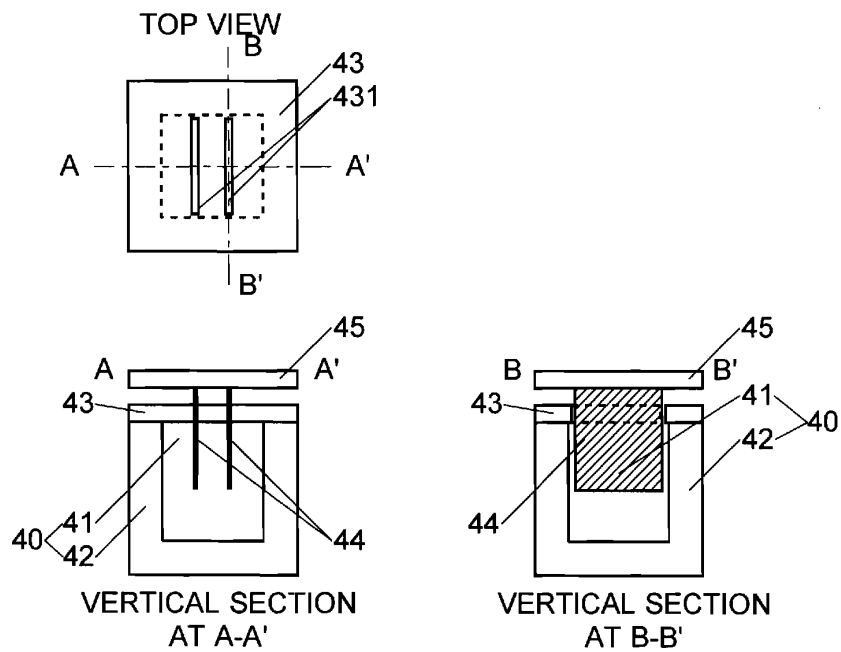
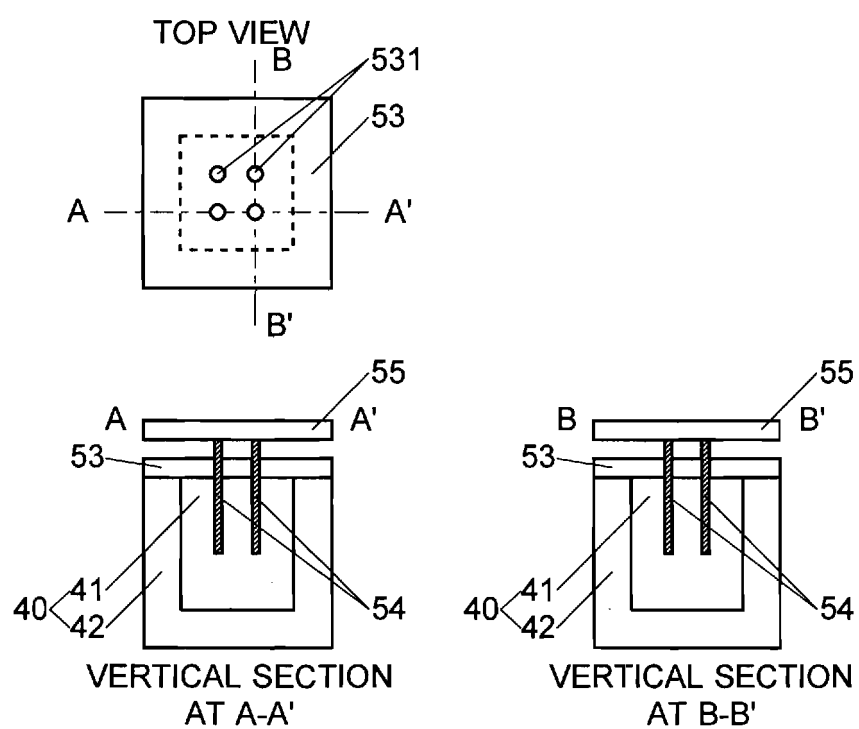


Fig. 11



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2009/005726

A. CLASSIFICATION OF SUBJECT MATTER

H01F41/02(2006.01)i, B22F3/02(2006.01)i, B22F3/24(2006.01)i, B22F5/10(2006.01)i, C22C28/00(2006.01)i, C22C33/02(2006.01)i, C22C38/00(2006.01)i, H01F1/053(2006.01)i, H01F1/08(2006.01)i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

H01F41/02, B22F3/02, B22F3/24, B22F5/10, C22C28/00, C22C33/02, C22C38/00, H01F1/053, H01F1/08

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

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2009
Kokai Jitsuyo Shinan Koho 1971-2009 Toroku Jitsuyo Shinan Koho 1994-2009

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.
X Y	JP 2007-134353 A (Intermetallics Co., Ltd.), 31 May 2007 (31.05.2007), paragraphs [0089], [0143]; fig. 8 (Family: none)	1-5, 10-11 1-11
X Y	JP 2001-78402 A (Shin-Etsu Chemical Co., Ltd.), 23 March 2001 (23.03.2001), paragraphs [0016] to [0017] & US 6606019 B1 & EP 1065777 A1	12 1-11
Y	JP 2007-53351 A (Shin-Etsu Chemical Co., Ltd.), 01 March 2007 (01.03.2007), claim 1; paragraphs [0033] to [0035], [0050] to [0051] & US 2007/0017601 A1 & EP 1746611 A1	7-8

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

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Date of the actual completion of the international search
22 December, 2009 (22.12.09)

Date of mailing of the international search report
12 January, 2010 (12.01.10)

Name and mailing address of the ISA/
Japanese Patent Office

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2009/005726

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2001-254104 A (Denso Corp.), 18 September 2001 (18.09.2001), entire text (Family: none)	1-12

Form PCT/ISA/210 (continuation of second sheet) (April 2007)

REFERENCES CITED IN THE DESCRIPTION

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