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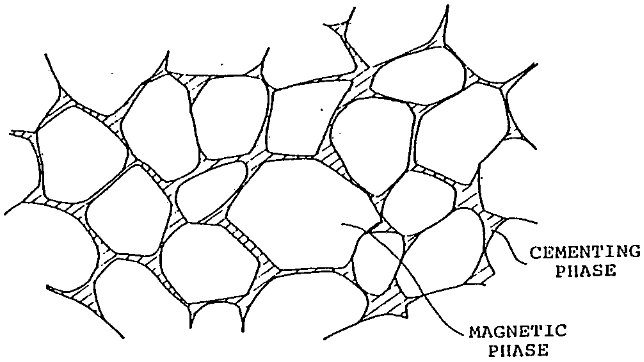
(54) Permanent magnetic material and method for producing the same.

(57) A permanent magnetic material of a desired bulk shape is obtained which comprises a composite microstructure consisting of magnetic particles of $Nd_2Fe_{14}B$ dispersed within a metallic cementing phase of 10% or less by volume of the magnetic material. A magnetic powder is prepared from an alloy comprising $Nd_2Fe_{14}B$ compound and is mixed with an alloy powder having a melting point lower than the peritectic temperature of $Nd_2Fe_{14}B$, and the mixed powder is sintered at a temperature higher than the melting point but lower than the peritectic temperature, so that the alloy powder melts and forms the cementing phase covering each magnetic particle to thereby realize high coercive force and excellent corrosion resistance. In place of Nd, other rare earth metal or metals can be used. A part of Fe can be replaced by other transition metal or metals. For the cementing metallic element or elements, Al, Zn, Sn, Cu, Pb, S, In Ga, Ge, and Te can be used.



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Permanent Magnetic Material and Method for Producing the same

1) Field of the Invention

This invention relates to a permanent magnet material of a bulk shape and, in particular, to an iron-rare earth metal-boron (R-Fe-B) permanent magnet material with a high coercive force.

2) Description of the Prior Art

Permanent magnets have been used in various applications such as electromechanical apparatus.

Recently, demands for Sm-Co permanent magnets have increased in place of known alnico magnets, ferrite magnets, and other conventional magnets, because of the high energy product of Sm-Co magnets. However, the Sm-Co magnets are expensive because of use of cobalt.

Therefore, various approaches are made for new permanent magnets which are economical and have an increased energy product.

A possible approach has been directed to a novel intermetallic compound of transition metal (T) and rare earth metal (R) instead of the Sm-Co intermetallic compound.

However, the intermetallic compounds without use of Co have been considered impossible to produce a magnet having coercivity which is associated with magnetocrystalline anisotropy because the compounds have an easy magnetization direction in the crystal phase. A reference is made to K. J. Strnat; IEEE Trans. Mag. (1972) 511.

In Appl. Phys. Lett. 39(10) (1981), 840, N. C. Koon and B. N. Das disclosed magnetic properties of amorphous and crystallized alloy of $(Fe_{0.82}B_{0.18})_{0.9}Tb_{0.05}La_{0.05}$. They wrote that crystallization of the alloy occurred near the relatively high temperature of 900 K, which also marked the onset of dramatic increase in the intrinsic coercive force. They found out that the alloy in the crystallized state appeared potentially useful as low cobalt permanent magnets.

It is considered that magnetically hard intermetallic compound of R-Fe-B (R = Tb and La) are formed in the alloy. Reviewing the R-Fe-B (R=Gd, Sn, Nd) ternary phase diagram by N. F. Chaban, Y. B. Kuz'ma,

N. S. Bilonizhko, O. O. Kachmar and N. W. petriv; Dopodivi Akad. Nuk. Ukr. RSR, Ser. A (1979) No.10, P.P. 875-877, the intermetallic compound R-Fe-B (R = Tb and La) by Koon et al is guessed to be represented by $R_3Fe_{16}B$, which is confirmed to be $Nd_2Fe_{14}B$ by J. J. Croat et al. Reference is made to J. J. Croat, J. F. Herbst, R. W. Lee and F. E. Pinkerton; J. Appl. Phys, 55 (1984) 2078.

Therefore, considering the saturation magnetization of an intermetallic compound of R-T as shown in the above-described reference by K. J. Strnat, it can be guessed that use of Ce, Pr, and/or Nd for R in Fe-B-R alloy can provide a better magnetic properties for permanent magnets than the Fe-B-La-Tb alloy.

J. J. Croat proposed amorphous (Nd and/or Pr)-Fe-B alloy having magnetic properties for a permanent magnet as disclosed in JP-A-60009852. Those magnetic properties was considered to be caused by a microstructure where $Nd_2Fe_{14}B$ particles having a perticle size of 20-30 nm were dispersed within an amorphous Fe phase. Reference is further made to R. K. Mishra: J. Magnetism and Magnetic Materials 54-57 (1986) 450.

However, the amorphous alloy can provide only an isotropic magnet because of its crystallographically isotropy. This means that a high performance permanent magnet cannot be obtained from the amorphous alloy.

Sagawa, Fujiwara, and Matsuura proposed an anisotropic R-Fe-B sintered magnet in JP-A-59046008 which was produced from an ingot of an alloy of R (especially Nd), Fe, and B by a conventional powder metallurgical processes. The sintered magnet has more excellent magnetic properties for permanent magnets than the known Sm-Co magnets.

However, the R-Fe-B sintered magnet has a problem in considerably low corrosion resistance.

Further, since the R-Fe-B alloy out of stoichiometric compound of $Nd_2Fe_{14}B$ is active to oxygen, any care is necessary so as to prevent the alloy from oxidation. Moreover a surface oxide layer must be machined away after sintering process. Therefore, the magnet cannot be produced with a high dimensional accuracy.

It is an object of the present invention to provide a permanent magnet material of a bulk shape comprising $R_2T_{14}B$ magnetic compound and having an excellent corrosion resistance.

It is another object of the present invention to provide a method for producing the bulk-shape permanent magnet material comprising $R_2T_{14}B$ magnetic compound and having an excellent corrosion resistance.

It is still another object of the present invention to provide a method for producing the bulk-shape permanent magnet material comprising $R_2T_{14}B$

magnetic compound with an excellent dimensional accuracy.

According to the present invention, a magnetic body with a high coercive force for a permanent magnet can be obtained which consists essentially of a metallic cementing phase and magnetic crystalline particles uniformly dispersed within the metal cementing phase. The cementing phase is 10% or less by volume of the magnetic body and comprises at least one element selected from a first metallic group of Al, Zn, Sn, Cu, Pb, S, In, Ga, Ge, and Te. The magnetic crystalline particles is substantially balance of the volume of the magnetic body and is a composition represented by a chemical formula $R_2T_{14}B$, where R is at least one element selected from Y and rare earth metals, T being transition metal and comprising Fe 50-100 at% in the transition metal. Each of the magnetic particles is embedded in the cementing phase to form an interface therebetween.

The cementing phase is inert to oxygen in comparison with Nd and has, therefore, a good corrosion resistance.

The cementing phase may comprise an intermetallic compound of at least one of the first metallic group and at least one selected from a second metallic group of R, T, and B.

The present invention further provides a method for producing a magnetic body with a high coercive force for a permanent magnet. The method comprises steps of: preparing an ingot of R-T-B magnetic alloy comprising a magnetic intermetallic compound represented by a chemical formula of $R_2T_{14}B$, where R is at least one element selected from Y and rare earth metals, T being transition metal but comprising Fe 50-100 at% in the transition metal; pulverizing and milling the ingot to thereby prepare a magnetic powder; preparing a metallic cementing powder comprising at least one element selected from a first metallic group of Al, Zn, Sn, Cu, Pb, S, In, Ga, Ge, and Te; mixing the metallic cementing powder of 10% or less by volume and the magnetic powder of substantially balance to prepare a mixed powder; and forming a bulk-shape body of the mixed powder at an elevated temperature.

The bulk-shape body forming step comprises steps of; compacting the mixed powder under influence of a grain aligning magnetic field into a compact body of a predetermined shape; and sintering the compact body at a temperature lower than a peritectic reaction temperature of the magnetic powder but higher than a melting temperature of the metallic cementing powder to thereby produce, as the bulk-shape body, a sintered body.

The sintered body may be subjected to a heat treatment at a temperature, preferably, 300-900 °C for improving the magnetic properties of the sintered body.

The bulk-shape body forming step may be hot compaction process for hot compacting the mixed powder into the bulk-shape body at an elevated temperature lower than 1,100 °C but higher than a melting temperature of the metallic cementing powder.

The hot compaction process is a hot-pressing process and alternatively a hot-extrusion process.

Fig. 1 is a schematic view of a microstructure of a bulk-form permanent magnet material according to the present invention;

Fig. 2 is a view illustrating variation of magnetic properties of a magnet to a volume percent of to $Nd_{30}Fe_{40}Al_{30}$ in the magnet;

Fig. 3 is a view illustrating a microstructure of a permanent magnet material of $(Nd_2Fe_{14}B)_{95}(Nd_{30}Fe_{40}Al_{30})_5$ according to an example of the present invention;

Fig. 4 is a view illustrating a microstructure of a permanent magnet material of $(Nd_2Fe_{14}B)_{89.5}(NdCu_2)_{10.5}$ according to another example of the present invention;

Fig. 5 is a view illustrating a microstructure of a permanent magnet material of $(Nd_2Fe_{14}B)_{95}(Pb_{69.6}Sn_{30.4})_5$ according to still another example of the present invention;

Fig. 6 is a view illustrating a microstructure of a permanent magnet material of

$(Nd_2Fe_{14}B)_{95}(Nd_{40}Fe_{20}Pb_{20}Sn_{20})_5$ according to yet another example of the present invention;

Fig. 7 is a view illustrating variation of a coercive force of a permanent magnet material in an example to a heat treatment temperature; and

Fig. 8 is a view illustrating variation of a coercive force of a permanent magnet material in an example to a heat treatment time period.

The present invention attempts to provide a permanent magnet material with a high coercive force and a corrosion resistance by making the material to have a microstructure as shown in Fig. 1 where magnetic particles of stoichiometric intermetallic compound of $Nd_2Fe_{14}B$ are dispersed within a metallic cementing phase. The metallic cementing phase is composed of metallic elements and/or an intermetallic compound or compounds, which are inert to oxygen in comparison with Nd.

In order to realize the microstructure, $Nd_2Fe_{14}B$ powder and metallic cementing powder are separately prepared and those powder are mixed. The mixed powder is sintered or hot-formed at an elevated temperature into a bulk-shape body so that the cementing powder forms a cementing phase to cement the magnetic particles together.

The metallic element or elements are selected to be ones each having a melting point lower than a peritectic temperature of the intermetallic compound of $Nd_2Fe_{14}B$, and the elevated temperature is also selected lower than the peritectic temperature but higher than the melting point of the cementing powder. Therefore, only the cementing powder melts to form the cementing phase by the sintering process or hot compaction process while the intermetallic compound of the magnetic powder is not melted but dispersed within the cementing phase.

Nd is active to oxygen if they do not form any intermetallic compound together or with other metallic element or elements. Therefore, it is desired that the cementing phase includes no Nd.

However, at the high temperature for the sintering or hot-compaction, Nd and/or Fe diffuse from the magnetic powder into the cementing phase so that the intermetallic compound of $Nd_2Fe_{14}B$ is decomposed. In order to prevent the intermetallic compound of each magnetic particle from decomposition, Nd and Fe are included in the cementing powder in the form of an intermetallic compound or compounds with other metallic element or elements. Alternatively, the magnetic powder is prepared as an alloy powder including Nd and Fe in addition to the intermetallic compound of $Nd_2Fe_{14}B$.

In either use of the former and the latter ways, Nd and Fe are present in the cementing phase of the produced body. However, Nd forms an intermetallic compound with the other metallic element or elements.

Accordingly, the cementing phase is also excellent in corrosion resistance.

Generally speaking, since a comparatively high temperature is used in the sintering temperature, both of the former and the latter can be employed. However, since a comparatively low temperature is used in the hot compaction process, the latter is employed because intermetallic compound of Nd and Fe with the other metallic element is apt to have a melting temperature higher than the temperature for hot compaction.

The amount of the cementing powder is limited at maximum 10 % by volume of the mixed powder, because when the cementing powder exceeds 10 vol%, the amount of the magnetic powder is insufficient to obtain a high remanence.

It should be noted that the intermetallic compound of $Nd_2Fe_{14}B$ has a high residual magnetic flux density but has not a coercive force sufficient for a permanent magnet.

Now, the present invention will be described in connection with examples.

Example 1

An ingot of an intermetallic magnetic alloy represented by $Nd_{13}Fe_{81}B_6$ was prepared by the induction melting in argon gas atmosphere. Purity factors of used start materials of Nd, Fe (electrolytic iron), and B were 98%, 99.9%, and 99.5% or more, respectively. The ingot was pulverized by a crusher to have a particle size below 24 mesh (Tyler) and, thereafter, finely divided by a ball mill into a fine magnetic powder having an average particle size of 3 μm.

On the other hand, various metallic powders (Zn, Al, S, In, Ga, Ge, Sn, Te, Cu, and Pb) for the cementing phase material were prepared, each powder having an average particle size of several tens micron meters and a purity of 99.9% or more.

Each metallic powder was mixed with the fine magnetic powder to form mixed powder. Each metal was adjusted at 5% by volume of the mixed powder. Zn, Al, S, In, Ga, Ge, Sn, Te, Cu, and Pb were, by weight, 5%, 2%, 2%, 5%, 4%, 4%, 5%, 4%, 6%, and 7% in the mixed powders, respectively.

After being mixed in a ball mill, each mixed powder was hot-pressed into a desired bulk-shape body under a pressing stress of 1,000 Kg.f/cm^2 at a temperature of 600°C within argon gas atmosphere for 15 minutes.

Each hot-pressed body was measured as to density d, residual magnetic flux density Br, coercive force ${}_IH_C$, maximum energy product (BH)max. The measured data are demonstrated with sample numbers 1-10 in Table 1.

As a comparative sample, starting materials of Nd, Fe, B, and Al were blended with each other to obtain an alloy represented by $(Nd_{15}Fe_{77}B_7)_{95}Al_5$ and an ingot of the alloy was produced by use of an induction furnace. The ingot was finely ground into a fine powder, which was, in turn, hot-pressed into a desired bulk-shape body under similar conditions as described above. While, an ingot represented by $Nd_{13}Fe_{81}B_6$ was prepared in the similar manner and a hot-pressed body was produced from the ingot under the similar manner as described above. Densities and magnetic properties of those two hot-pressed bodies are shown with sample numbers 11 and 12, respectively, in Table 1.

Table 1

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
1	$(Nd_{13}Fe_{81}B_6)_{95}Zn_5$	7.5	7.1	10.8	9.8
2	$(Nd_{13}Fe_{81}B_6)_{95}Al_5$	7.3	7.0	12.6	10.1
3	$(Nd_{13}Fe_{81}B_6)_{96}S_4$	7.1	6.8	10.5	9.2
4	$(Nd_{13}Fe_{81}B_6)_{97}In_3$	7.5	6.7	9.9	8.6
5	$(Nd_{13}Fe_{81}B_6)_{96}Ga_4$	7.4	6.9	10.6	9.4
6	$(Nd_{13}Fe_{81}B_6)_{96}Ge_4$	7.4	6.8	10.0	8.9
7	$(Nd_{13}Fe_{81}B_6)_{97}Sn_3$	7.5	6.6	10.1	8.5
8	$(Nd_{13}Fe_{81}B_6)_{98}Te_2$	7.4	7.0	7.9	9.1
9	$(Nd_{13}Fe_{81}B_6)_{94}Cu_6$	7.6	6.7	9.1	8.3
10	$(Nd_{13}Fe_{81}B_6)_{98}Pb_2$	7.7	6.7	9.2	8.2
11*	$(Nd_{16}Fe_{77}B_7)_{95}Al_5$	7.2	6.3	9.6	7.5
12*	$Nd_{13}Fe_{81}B_6$	7.5	1	0.2	1

* Sample according to prior art

Table 1 teaches that the samples Nos. 1-10 according to an embodiment of this invention are superior to comparing samples Nos. 11 and 12 according to the known production method in the magnetic properties, especially, the maximum energy product (BH)max.

Example 2

A fine magnetic powder of $Nd_{13}Fe_{81}B_6$ was prepared and was mixed with metal powders (each having an average particle size of 20-30 um) of Zn 5wt%, Al 2wt%, S 2wt%, In 5wt%, Ga 4wt%, Ge 4wt%, Sn 5wt%, Te 4wt%, Cu 6wt%, and Pb 7wt%, respectively, in the similar manner as described in Example 1.

Each mixed powder was compacted into a compact body of a desired bulk-shape by application of a pressing force of 1.5 ton.f/cm^2 under influence of a grain aligning magnetic field of 25 KOe. The compact body was hot-pressed at 600°C by pressing stress of 1,000 Kg.f/cm^2 within vacuum for 15 minutes. Each hot-pressed body was subjected to measurement of density and magnetic properties. The measured data are shown as sample Nos. 13-22 in Table 2.

For comparing with these samples, alloy ingots of $(Nd_{15}Fe_{77}B_7)_{95}Al_5$ and $Nd_{14}Fe_{81}B_6$ were produced and were finely ground into powders. Those powders were compacted into compact bodies of a desired bulk shape and were hot-pressed in the similar manner. Densities and magnetic properties of those hot-pressed bodies are shown as sample Nos. 23 and 24 in Table 2.

Table 2

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
13	$(Nd_{13}Fe_{81}B_6)_{95}Zn_5$	7.5	13.5	11.1	42.0
14	$(Nd_{13}Fe_{81}B_6)_{95}Al_5$	7.3	14.0	12.9	45.3
15	$(Nd_{13}Fe_{81}B_6)_{96}S_4$	7.1	13.9	10.6	41.9
16	$(Nd_{13}Fe_{81}B_6)_{97}In_3$	7.5	13.6	10.3	40.1
17	$(Nd_{13}Fe_{81}B_6)_{96}Ga_4$	7.4	13.7	11.1	41.2
18	$(Nd_{13}Fe_{81}B_6)_{96}Ge_4$	7.4	13.7	10.4	39.8
19	$(Nd_{13}Fe_{81}B_6)_{97}Sn_3$	7.5	13.6	10.6	40.0
20	$(Nd_{13}Fe_{81}B_6)_{98}Te_2$	7.4	13.8	7.8	39.1
21	$(Nd_{13}Fe_{81}B_6)_{94}Cu_6$	7.6	13.4	9.3	39.6
22	$(Nd_{13}Fe_{81}B_6)_{98}Pb_2$	7.7	13.3	9.2	38.7
23*	$(Nd_{16}Fe_{77}B_7)_{95}Al_5$	7.2	9.8	6.4	22.8
24*	$Nd_{13}Fe_{81}B_6$	7.5	1	0.2	1

* Sample according to prior art

It should be noted from Table 2 that sample Nos. 13-22 according to embodiments of the present invention are superior in the magnetic properties to not only comparing samples of Nos 23 and 24 according to the known producing method but also samples of Nos. 1-10 in

Table 1 according to embodiments of the present invention.

Example 3

In the similar manner as described in Example 1, $Nd_2Fe_{14}B$ magnetic alloy ingot was prepared and was pulverized to have a particle size below 24 mesh (Tyler).

While, for the cementing phase material, start materials of Nd (having a purity factor of 98% or more), Fe (having a purity factor of 99.9% or more), and Al (having a purity factor of 99.9%) were mixed and melted by the induction melting in argon gas atmosphere to form an ingot of $Nd_{30}Fe_{40}Al_{30}$ alloy. The ingot was also pulverized into powder having a particle size below 24 (Tyler) mesh.

Both of the powders were blended with each other at various mixing ratios so that $Nd_{30}Fe_{40}Al_{30}$ powder is 0-15% by volume of the blended powder.

Each blended powder was finely ground into a powder of an average particle size of about 4 μm. The finely ground powder was compacted into a compact body of a desired bulk-shape by application compacting stress of 1.5 ton.f/cm^2 under influence of a grain aligning magnetic field of 20 KOe. The compact body was sintered in vacuum at 1,000-1,150 $^{\circ}$C for 2 hours. The sintered body was heat treated at 500-900 $^{\circ}$C for one hour. Density d, residual magnetic flux density Br, coercive force $_IH_C$, and (BH)max of the sintered body after heat treatment were measured and are illustrated in Fig. 2 for various volume percents of $Nd_{30}Fe_{40}Al_{30}$ in the sintered body.

In Fig. 2, the maximum magnetic properties are obtained at 5 vol% of $Nd_{30}Fe_{40}Al_{30}$ content. The magnetic properties are shown as a sample No. 25 in Table 3. The table also has properties of a comparing sample No. 26 which was a sintered $Nd_{14}Fe_{80}B_6$ alloy produced from the alloy ingot through milling, compacting in the aligning magnetic field, sintering, and heat treating steps according to a conventional powder metallurgy.

Table 3

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
25	$(Nd_2Fe_{14}B)_{95}\cdot(Nd_{30}Fe_{40}Al_{30})_5$	7.4	14.5	15.0	48.5
26*	$Nd_{14}Fe_{80}B_6$	7.5	12.8	10.0	38.0

* Sample according to prior art

It will be understood from Table 3 that sample No.25 according to the present invention has a considerably excellent magnetic properties in comparison with sample No. 26 according to the conventional process.

The sintered body of sample No. 25 was cross-sectioned and polished. The microstructure in the polished surface was observed by a Scanning Electron Microscope (SEM). The observed microstructure is illustrated in Fig. 3.

Referring to Fig. 3, it will be noted that the magnetic crystalline particles (being black) of $Nd_2Fe_{14}B$ are covered with, or embedded in, a cementing phase (being white) of $Nd_{30}Fe_{40}Al_{30}$.

Then, the No.25 sample was subjected to component analysis of the magnetic particles and the cemented phase in the sample by use of an Electron Probe Micro Analyser (EPMA). Analysed elements and their amount (at%) are shown in Table 4.

Table 4

Phase	Nd	Fe	Al	B	Composition
Cementing	25.2	70.2	4.6	0	$Nd(FeAl)_{1-3}$
magnetic	12.3	81.8	0	6	$Nd(FeAl)_{14}B$

This example teaches us that magnetic properties of sintered $Nd_2Fe_{14}B$ magnet can be considerably improved by covering and cementing the $Nd_2Fe_{14}B$ particles with the $Nd(Fe, Al)_3$ matrix or the cementing phase.

Fig. 2 shows that the magnetic properties gradually decrease along increase of the cementing phase from 5 vol% through 8 vol%. It is considered that this is because of decrease of the amount of the magnetic particles.

Example 4

$Nd_2Fe_{14}B$ alloy powder and $Nd_{30}Fe_{40}Al_{30}$ alloy powder produced through the similar steps as described in Example 3 were blended with each other so that $Nd_{30}Fe_{40}Al_{30}$ powder was 5% of volume of the blended powder. The blended powder was compacted to a desired bulk shape in the grain aligning magnetic field of 20 KOe by application of compacting force of 1.5 ton.f/cm^2 and produced a green compact body. The green compact body was subjected to a hot compaction by hot-pressing the green compact at 800 $^{\circ}$C in argon gas atmosphere by 1,000 Kg.f/cm^2 for 15 minutes.

Density and magnetic properties of the hot-pressed body were measured and are described with sample number of No. 27 in Table 5.

In Table 5, sample No. 28 is a sample produced from a $Nd_{14}Fe_{80}B_6$ powder through the similar compacting and hot-pressing steps.

Comparison of sample Nos. 27 and 28 also teach that the Nd-Fe-Al cementing phase improves the magnetic properties of $Nd_2Fe_{14}B$ magnet.

Table 5

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
27	$(Nd_2Fe_{14}B)_{95}\cdot(Nd_{30}Fe_{40}Al_{30})_5$	7.5	14.2	16.0	47.5
28*	$Nd_{14}Fe_{80}B_6$	7.5	1.0	0.2	1

* Sample according to prior art

Example 5

$Nd_2Fe_{14}B$ magnetic alloy powder with an average particle size of 3 µm was prepared in the similar manner as described in Example 1. While, $NdCu_2$ powder for the cementing phase material having similar particle size was also produced in a similar producing method. Both powders were mixed with each other so that the amount of $NdCu_2$ powder was about 10% by volume in the mixture, and the mixture was uniformly mixed in a ball mill. The mixture was compacted into a bulk shape by application of compacting stress of 1.5 ton.f/cm^2 in the grain aligning magnetic field of 20 KOe. The compacted body was sintered in vacuum at 1,100-1,130 °C for 2 hours. The sintered body was heat-treated in argon gas atmosphere at 600-800 °C for one hour.

The magnetic properties of the sintered body as sample No. 29 are shown in Table 6 together with its density.

Table 6

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
29	$(Nd_2Fe_{14}B)_{89.5}\cdot(NdCu_2)_{10.5}$	7.5	12.8	9.3	38.5

No. 29 sample was then cross-sectioned and polished. The microstructure of the cross-section was observed by use of an optical microscope. The observed microstructure is illustrated in Fig. 4. In the figure, $Nd_2Fe_{14}B$ magnetic particle is shown in white and the $NdCu_2$ cementing phase is shown in black.

Further, the electron beam X-ray microanalysis was carried out to the magnetic particle phase and the cementing phase of the sample of No. 29. Table 7 shows analysed elements and their amounts by atomic percent (at%).

In Table 7, the cementing phase comprises two intermetallic compounds I and II. Compound I is represented by NdCu, and Compound II is represented by Nd(Cu, Fe)$_2$.

Table 7

Phase		Nd	Cu	Fe	B
Magnetic		13.1	-	81.8	6.0
Cementing	I	46.9	53.1	-	-
	II	31.3	60.8	7.9	-

As being illustrated in Fig. 4, each magnetic particle (shown in white in Fig. 4) is cemented and covered with the cementing phase (black in Fig. 4).

Example 6

A mixture similar to in Example 5 was prepared.

The mixture was hot-pressed in argon gas atmosphere at 900 °C by application of pressing stress of 1,000 Kg.f/cm^2 for 15 minutes.

Table 8

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
30	$(Nd_2Fe_{14}B)_{89.5}\cdot(NdCu_2)_{10.5}$	7.45	12.4	8.5	36.0

Table 8 shows density and magnetic properties of the hot-pressed body as sample No. 30. Table 8 teaches us that excellent magnetic properties are obtained.

Example 7

$Nd_2Fe_{14}B$ magnetic powder similar to Example 5 was prepared. While, $Nd_{25.4}Cu_{52.2}Zn_{22.4}$ powder for the cementing phase was prepared in the similar manner as the magnetic powder. Both of the powders were blended with each other so that the amount of the $Nd_{25.4}Cu_{52.2}Zn_{22.4}$ powder was about 10% by volume of the blended powder. The blended powder was uniformly mixed in ball mill. The mixture was hot-pressed into a body of a desired shape in argon gas at 600 ^{o}C by pressing stress of 1,000 $Kg.f/cm^2$ for 15 minutes.

The magnetic properties and density of the hot-pressed body are shown in Table 9.

Table 9

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
31	$(Nd_2Fe_{14}B)_{89.4}\cdot(Nd_{25.4}Cu_{52.2}Zn_{22.4})_{10.6}$	7.42	12.2	9.5	35.0

Example 8

A molten alloy of $Nd_{34}Fe_{65}B$ was prepared in the similar way as in Example 1.

While, two Pb-Sn alloys A and B shown in Table 10 were also prepared in the molten state by using start materials of Pb and Sn each having purity factor of 99.9% or more.

Table 10

Cementing Alloy	Pb	Sn
A (at%)	69.6	30.4
B (at%)	26.1	73.9

A powder less than 250 (Tyler) mesh was prepared from each of the molten alloys by atomization. Then, $Nd_{16}Fe_{18}B_6$ magnetic powder was divided by ball mill into average particle size of about 3 µm. Each powder of Pb-Sn alloys A and B was blended with the magnetic powder and was mixed in ball mill. The amount of each of alloy A and B was 5% by volume of each mixture.

Each powder mixture was compacted into a compact body of a bulk shape in the aligning magnetic field of 20 KOe by compacting stress of 1.5 ton.f/cm^2. The compact body was sintered in vacuum at 1,000-1,150 °C for 2 hours. The sintered body was heat-treated at

500-900 °C for one hour and was thereafter subjected to measurement of density and magnetic properties.

The measured data are demonstrated in Table 11. No. 32 sample uses alloy A and No. 33 uses alloy B.

Table 11

Sample No.	Cementing Alloy	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
32	A	7.5	12.9	11.5	38.0
33	B	7.5	12.8	12.0	37.5

The microstructure of each sample of Nos. 32 and 33 was observed by an optical microscope. Fig. 5 shows the microstructure of No. 32 sample. In the figure, the magnetic particles are illustrated in white and the cementing phase is black.

While, the microanalysis was performed to the magnetic perticle phase and the cementing phase of each sample of Nos. 32 and 33. The analysed elements are shown in Table 12 together with their amounts by at%.

Attention should be directed to a fact that the magnetic particle phase consists of the intermetallic compound of $Nd_2Fe_{14}B$. While, Nd and Fe is present in both of the cementing phase. I and II are analysed data at different portions of the cementing phase of the sintered body.

Table 12

Sample	Phase		Nd	Fe	B	Pb	Sn	formula
32	Magnetic		12.3	81.7	6	-	-	$Nd_2Fe_{14}B$
	Cementing	I	50.5	26.0	-	17.5	6.0	Nd(Fe, Pb,Sn)
		II	53.0	20.0	-	22.0	5.0	
33	Magnetic		12.6	81.2	6.2	-	-	$Nd_2Fe_{14}B$
	Cementing	I	48.5	27.0	-	6.5	18.0	Nd(Fe, Pb,Sn)
		II	52.0	34.0	-	5.0	9.0	

For sample No. 33, it will be appreciated that the cementing phase is constituted of an intermetallic compound of Nd(Pb, Fe, Sn) where a part of Pb in NdPb is replaced by Fe and Sn. While, the cementing phase in No. 33 sample is composed of an intermetallic compound of Nd(Sn, Fe, Pb) where a part of Sn in NdSn compound is replaced by Fe and Pb.

That is, Nd and Fe diffuse from Nd-Fe-B particles into Pb-Sn phase so that each Nd-Fe-B particle becomes a stoichiometric intermetallic compound $R_2Fe_{14}B$ which has ferromagnetism.

If Nd and Fe are insufficiently diffused in the Pb-Sn alloy, excellent magnetic properties are not obtained because each magnetic particle comprises not only the intermetallic compound of $Nd_2Fe_{14}B$ but also non-magnetic phase.

This means that it is useful for production of the permanent magnet material to prepare $Nd_2Fe_{14}B$ particles and to mix the particles with cementing phase materials so as to obtain a bulk-shape magnet material having excellent magnetic properties.

Example 9

The powder mixtures prepared in Example 8 were compacted into green compact bodies in the aligning magnetic field of 20 KOe by compacting stress of 1.5 ton.f/cm^2. Each green compact body was hot-pressed in argon gas at 750 ^{o}C under application of a pressure 1,000 Kg.f/cm^2 for 20 minutes. Thereafter, the hot-pressed body was heat-treated in argon gas at 600 ^{o}C for 30 minutes.

The magnetic properties after the heat treatment are shown in Table 13. Samples of Nos. 34 and 35 in Table 13 contain the Pb-Sn alloys A and B in Table 10, respectively.

Table 13

Sample No.	Alloy No.	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
34	A	7.4	12.5	12.0	36.5
35	B	7.4	12.4	12.5	36.0

In this embodiment, it was also confirmed that Nd and Fe were present in the cementing phase with the particles becoming $Nd_2Fe_{14}B$ compound.

Example 10

$Nd_2Fe_{14}B$ magnetic alloy powder similar to Example 3 was prepared.

In the similar producing steps, Nd-Fe-Pb-Sn alloy powders C, D, and E in Table 14 were produced from

Table 14

Cementing Alloy	Nd	Fe	Pb	Sn
C	40	20	20	20
D	40	10	30	20
E	40	10	20	30

start materials of Nd (purity of 98 % or more), Fe (purity of 99.9% or more), Pb (purity of 99.9%), and Sn (purity of 99.9%). Each powder of C-E was prepared to have an average particle size of 3 µm.

These powders C-E were blended with the magnetic powder, separately, and each blended powder was mixed with each other in a ball mill. Each alloy powder of C-E was 5% by volume of each powder mixture.

Each powder mixture was compacted into a compact body in the aligning magnetic field of 20 KOe by compacting force of 1.5 ton.f/cm^2, and the compact body was sintered in vacuum at 1,000-1,150 °C for 2 hours. The sintered body was further heat-treated at 500-900 °C.

Table 15 indicates density and magnetic properties of each sintered and heat-treated body as samples Nos. 36, 37, and 38 which use Nd-Fe-Pb-Sn alloys C, D, and E, respectively.

Table 15

Sample No.	Cementing Alloy	d (g/cm^3)	Br (KG)	$_I H_C$ (KOe)	(BH)max (MGOe)
36	C	7.5	12.9	11.4	37.5
37	D	7.6	12.8	10.9	37.5
38	E	7.5	12.9	11.0	38.5

In comparison with Nos. 32 and 33 samples, Nos. 36-38 samples are nearly equal to them in the magnetic properties but have a reduced Nd content which is active to oxygen. Accordingly, Nos. 36-38 samples are superior to Nos. 32 and 33 samples in the corrosion resistance and especially during production and storage of powder materials.

Table 16

Sample	Phase	Nd	Fe	B	Pb	Sn
36	Magnetic	12.2	81.8	6.0	-	-
	Cementing	41.5	19.7	-	21.0	17.8
37	Magnetic	12.4	81.7	5.9	-	-
	Cementing	40.2	9.3	-	30.8	19.7
38	Magnetic	12.5	81.4	6.1	-	-
	Cementing	40.8	10.1	-	20.8	18.3

For each sintered body, the observation of microstructure and microanalysis were carried out. The microstructure of No. 38 sample is shown in Fig. 6

(white for magnetic particles and black for cementing phase), and analysed elements in respective samples are described in Table 16.

It will be understood from Table 16 that not only B but Nd and Fe do not diffuse into the Nd-Fe-Pb-Sn cementing phase. Pb and Sn nor diffuse into $Nd_2Fe_{14}B$ particles.

Example 11

Each mixture obtained in Example 10 was compacted into a green compact in the aligning magnetic field of 25 KOe by pressing stress of 1.5 ton.f/cm^2. The green compact was hot-pressed in argon gas at 800 °C by pressing stress of 1.0 ton.f/cm^2 for 20 minutes. Then, the hot-pressed body was heat-treated in argon gas atmosphere at 600 °C for 30 minutes.

Table 17 shows density and magnetic properties of each hot-pressed body after heat-treated as samples Nos. 39, 40, and 41.

Table 17

Sample No.	Cementing Alloy	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
39	C	7.5	12.9	11.0	36.5
40	D	7.6	12.4	9.8	35.0
41	E	7.5	12.5	11.5	36.0

Nos. 39 and 40 samples have magnetic properties equally to Nos. 34 and 35 samples but have a reduced amount of Nd.

Next, in order to appreciate of corrosion resistance of permanent magnet material according to the present invention, samples of Nos. 25, 29, and 32 according to the present invention and No. 26 sample sintered by the conventional powder metallurgy were subjected to the following tests.

Test 1

A humidity test was carried out onto the test pieces under a test condition of a temperature of 60 °C and a humidity of 90% for 100 hours.

Table 18

Sample	90% Humidity Test At 60 °C							
No.	Time(Hr.)	1	5	10	25	50	75	100
29		G	G	G	X	Y	Z	Z
32		G	G	G	G	X	Y	Z
25		G	G	G	G	G	X	Y
26		G	X	X	Y	Z	Z	Z

G ...No change.

X ...White oxide slightly observed.

Y ...Red rust occurred on the surface.

Z ...Red rust presented over the entire surface.

The result of the test is shown in Table 18, which teaches us that the samples according to the present invention are superior in corrosion resistance to the sample according to the conventional powder metallurgy.

Test 2

Test pieces of each sample were coated with surface coatings by electrolytic Ni plating, phosphate treating, and anti-corrosion resin coating, respectively.

The electrolytic Ni plating was carried out after Cu plating coating of 3-5 µm thickness was previously formed.

The phosphate treating was performed using a conventional phosphate solution.

In the anti-corrosion resin coating, epoxy resin was dissolved in an organic solvent and then sprayed onto test pieces. Thereafter, epoxy resin coating was formed by heating at 150 °C.

Those test pieces were subjected to salt spray corrosion test under a condition where salt water solution (5% NaCl) was sprayed onto the test pieces at 35 °C for 48 hours during which surface change of each test piece was observed. When the test completed, magnetic properties of each test were measured.

The observed surface change and the measured magnetic properties are illustrated in Tables 19 and 20.

It will be noted from Table 19 that permanent magnet materials according to the present invention are excellent in corrosion resistance. The magnetic properties are not degraded by anti-corrosion surface coating as well be understood from Table 20.

Table 19

Coating		Salt Spray Corrosion Test				
Kind	Thickness (μm)	Time (Hr.)	Sample Number 29	32	25	26
Ni plate	10-20	12	G	G	G	Z
Phosphate	5-7	3	X	X	G	Z
Epoxy resin	15-25	24	G	G	G	Z

G ...No change.

X ...White oxide slightly observed.

Y ...Red rust occurred on the surface.

Z ...Red rust presented over the entire surface.

Table 20

Sample		Coating		
	Kind	Ni plate	Phosphate	Epoxy resin
	Thick.	10-20 μm	5-7 μm	15-25 μm
29	Br (KG)	14.1	14.2	14.0
	$_IH_C$ (KOe)	14.8	15.3	14.5
	(BH)max (MGOe)	46.9	47.1	46.8
32	Br (KG)	12.8	12.4	12.6
	$_IH_C$ (KOe)	9.4	9.0	9.8
	(BH)max (MGOe)	38.5	35.4	36.5
25	Br (KG)	12.8	12.9	12.6
	$_IH_C$ (KOe)	11.0	12.0	11.9
	(BH)max (MGOe)	37.0	37.6	36.5

Example 12

Zn, Sn, and Al powders, each having a purity factor of 99.9%, were blended with the $Nd_{13}Fe_{81}B_6$ magnetic alloy powder obtained in Example 2, respectively, and the blend was mixed uniformly in ball mills to obtain respective powder mixtures.

Added amounts of Zn, Sn, and Al powders were selected to be 5% by volume of respective powder mixtures.

Each powder mixture was compacted in the aligning magnetic field of 25 KOe by pressing stress of 1.5 ton.f/cm^2 and formed a green compact of a desired bulk shape. The green compact was heated at 700 ^{o}C and then coated with glass coating by depositing glass powder onto the surface of the green compact. The glass coated green compact was inserted in a extrusion mould and was moulded into a desired shape by extrusion.

Each extruded body was measured to obtain the magnetic properties. The measured properties and densities are indicated for Nos. 42, 43, and 44 samples in Table 21 together with density.

Table 21

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_{I}H_{C}$ (KOe)	(BH)max (MGOe)
42	$(Nd_{13}Fe_{81}B_{6})_{96}Al_{4}$	7.3	13.8	9.9	41.2
43	$(Nd_{13}Fe_{81}B_{6})_{95}Zn_{5}$	7.5	13.4	5.8	37.8
44	$(Nd_{13}Fe_{81}B_{6})_{97}Sn_{3}$	7.5	13.3	7.0	37.0
45*	$(Nd_{13}Fe_{81}B_{6})_{89}Al_{11}$	7.2	9.8	6.4	22.8
46*	$Nd_{13}Fe_{81}B_{6})$	7.5	1.0	0.2	1

*...Sample according to the Prior Art

In Table 21, each of Nos. 45 and 46 samples is a hot-pressed body produced from the alloy ingot through pulverizing, compacting, and extruding steps.

Magnetic materials according to the present invention can be produced by hot extrusion and has excellent magnetic properties in comparison with one produced from the alloy ingot.

Example 13

Each of green compact obtained in Example 12 was hot-pressed in argon gas atmosphere at 700 °C by pressing stress of 1,000 Kg.f/cm^2 for 15 minutes. The hot-pressed body was heat-treated in argon at 600 °C for 10 minutes.

Table 22

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
47	$(Nd_{13}Fe_{81}B_6)_{96}Al_4$	7.3	14.9	10.1	49.7
	After Heat Treatment	7.3	14.1	20.3	46.9
48	$(Nd_{13}Fe_{81}B_6)_{95}Zn_5$	7.3	13.5	6.1	39.8
	After Heat Treatment	7.3	13.3	18.9	41.8
49	$(Nd_{13}Fe_{81}B_6)_{97}Sn_3$	7.5	13.6	10.6	44.2
	After Heat Treatment	7.5	13.4	16.6	42.4

Magnetic properties of each hot-pressed body as sample of Nos. 47, 48, and 49 were measured before and after the heat treatment and are illustrated in Table 22. It will be found out from Table 22 that the coercive force ${}_{I}H_{C}$ is improved by the heat-treatment with the other properties being almost unchanged. The improvement is considered to be caused due to decrease of nucleation sites of reverse domain by the heat-treatment.

In order to confirm influence of heat-treating temperature for the improvement of coercive force, heat treatment was carried out to No. 47 sample at various temperatures. Fig. 7 illustrates measured coercive force to variation of heat-treating temperature. Fig. 7 shows that the effective temperature is 300-900 °C.

Further, experiment for heat-treatment was performed as to No. 47 sample at various heat-treating time periods at a constant temperature of 600 °C. Fig. 8 shows residual magnetic flux density Br and coercive force ${}_{I}H_{C}$ to variation of heat-treating time period. It will be noted from Fig. 8 that the heat treatment for about 10 minutes is most effective for improvement of the magnetic properties.

Example 14

$Al_{11}Zn_{89}$ powder and $Al_{83}Cu_{17}$ powder were produced from starting materials having purity of 98% or more. Each powder has an averaged particle size of 1-10 μm.

Each powder and the $Nd_{13}Fe_{81}B_6$ magnetic powder obtained in Example 2 were mixed with each other at a mixing ratio of 5 to 95 by volume (which means 4.4 wt% for $Al_{11}Zn_{89}$ and 2.4 wt% for $Nd_{13}Fe_{81}B_6$) in a ball mill.

Table 23

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
50	$(Nd_{13}Fe_{81}B_6)_{95}\cdot(Al_{11}Zn_{89})_5$	7.4	13.4	10.2	40.8
51	$(Nd_{13}Fe_{81}B_6)_{95}\cdot(Al_{83}Cu_{17})_5$	7.3	13.5	9.5	42.3

The mixture was compacted to a green compact in the aligning magnetic field of 25 KOe by pressing stress of 1.5 ton.f/cm^2. The green compact was hot-pressed in argon gas atmosphere at 600 ^{o}C by pressing stress of 1,000 Kg.f/cm^2 for 15 minutes. The magnetic properties of hot-pressed body of sample Nos. 50 and 51 are described in Table 23.

It will be understood to obtain improvement of magnetic properties similar to Example 2.

Example 15

$Al_{66}Fe_{34}$ powder, $Al_{25}Ni_{75}$ powder, $Al_{20}Co_{80}$ powder, and $Al_{75}Cr_{25}$ powder were used in place of $Al_{11}Zn_{89}$ powder and $Al_{83}Cu_{17}$ powder in Example 14, and a green compact was produced from a mixture of each powder and $Nd_{13}Fe_{81}B_6$ magnetic powder in similar manner as described in Example 14. The green compact was hot-pressed similarly, but for 10 minutes.

Table 24

Sample No.	Composition	d (g/cm^3)	Br (KG)	$_IH_C$ (KOe)	(BH)max (MGOe)
52	$(Nd_{13}Fe_{81}B_6)_{95}\cdot(Al_{66}Fe_{34})_5$	7.3	13.2	9.5	39.8
53	$(Nd_{13}Fe_{81}B_6)_{94}\cdot(Al_{25}Ni_{75})_6$	7.4	13.0	9.1	39.5
54	$(Nd_{13}Fe_{81}B_6)_{94}\cdot(Al_{20}Co_{80})_6$	7.5	13.7	9.4	42.3
55	$(Nd_{13}Fe_{81}B_6)_{95}\cdot(Al_{75}Cr_{25})_5$	7.3	13.6	9.8	42.5

Magnetic properties of the hot-pressed body was excellent similarly as in Example 14 and is shown in Table 24 with sample numbers Nos. 52-55 for alloy powders used.

CLAIMS:

1. A magnetic body with a high coercive force for a permanent magnet which consists essentially of a metallic cementing phase and magnetic crystalline particles uniformly dispersed within said metal cementing phase, said cementing phase being 10% or less by volume of said magnetic body and comprising at least one element selected from a first metallic group of Al, Zn, Sn, Cu, Pb, S, In, Ga, Ge, and Te, said magnetic crystalline particles being substantially balance of the volume of the magnetic body and being a composition represented by a chemical formula $R_2T_{14}B$, where R is at least one element selected from Y and rare earth metals, T being transition metals and comprising Fe 50-100 at% in the transition metal, each of said magnetic particles being embedded in said cementing phase to form an interface therebetween.

2. A magnetic body as claimed in Claim 1, said cementing phase comprising an intermetallic compound of at least one of said first metallic group and at least one selected from a second metallic group of R, T, and B.

3. A method for producing a magnetic body with a high coercive force for a permanent magnet comprising steps of:

preparing an ingot of R-T-B magnetic alloy comprising a magnetic intermetallic compound

(Claim 3 continued)

represented by a chemical formula of $R_2T_{14}B$, where R is at least one element selected from Y and rare earth metals, T being transition metal but comprising Fe 50-100 at% in the transition metal;

pulverizing and milling said ingot to thereby prepare a magnetic powder;

preparing a metallic cementing powder comprising at least one element selected from a first metallic group of Al, Zn, Sn, Cu, Pb, S, In, Ga, Ge, and Te;

mixing a metallic cementing powder of 10% or less by volume and said magnetic powder of substantially balance to prepare a mixed powder; and

forming a bulk-shape body of said mixed powder at an elevated temperature.

4. A method as claimed in Claim 3, said bulk-shape body forming step comprising;

compacting said mixed powder under influence of a grain aligning magnetic field into a compact body of a predetermined shape; and

sintering said compact body at a temperature lower than a peritectic reaction temperature of said magnetic powder but higher than a melting temperature of said metallic cementing powder to thereby produce, as said bulk-shape body, a sintered body.

5. A method as claimed in Claim 4, wherein said R-T-B ingot consists of said intermetallic compound of $R_2T_{14}B$, and said metallic cementing powder comprises an

(Claim 5 continued)

intermetallic compound of at least one element selected from said first metallic group and at least one selected from a second metallic group of R, T, and B.

6. A method as claimed in Claim 5, further comprising a step of subjecting said sintered body to a heat treatment for improving the magnetic properties of the sintered body.

7. A method as claimed in Claim 6, wherein said heat treatment is performed at 300-900 °C.

8. A method as claimed in Claim 3, wherein said bulk-shape body forming step is a hot compaction process for hot compacting said mixed powder into said bulk-shape body at an elevated temperature lower than 1,100 °C but higher than a melting temperature of said metallic cementing powder.

9. A method as claimed in Claim 8, wherein said hot compaction process is a hot pressing process for hot pressing said mixed powder into said bulk-shape body under a pressure of 5-5,000 Kg.f/cm^2.

10. A method as claimed in Claim 9, said bulk shape body forming step further comprising a step of previously compacting said mixed powder to form a green compact, said green compact being then subjected to said hot-pressing process.

11. A method as claimed in Claim 10, wherein said compacting step is performed within an aligning magnetic field.

12. A method as claimed in Claim 8, wherein said hot compaction process comprises steps of compacting said mixed powder to form a green compact, heating said green compact at said elevated temperature, and then subjecting said heated green compact to extrusion to thereby produce, as said bulk-shape body, an extruded body.

13. A method as claimed in Claim 12, wherein said green compact is coated with a heat insulating material prior to said extrusion.

14. A method as claimed in Claim 8, further comprising a step of subjecting said bulk-shape body to a heat treatment for improving the magnetic properties thereof.

15. A method as claimed in Claim 14, wherein said heat treatment is performed at 300-900 °C.

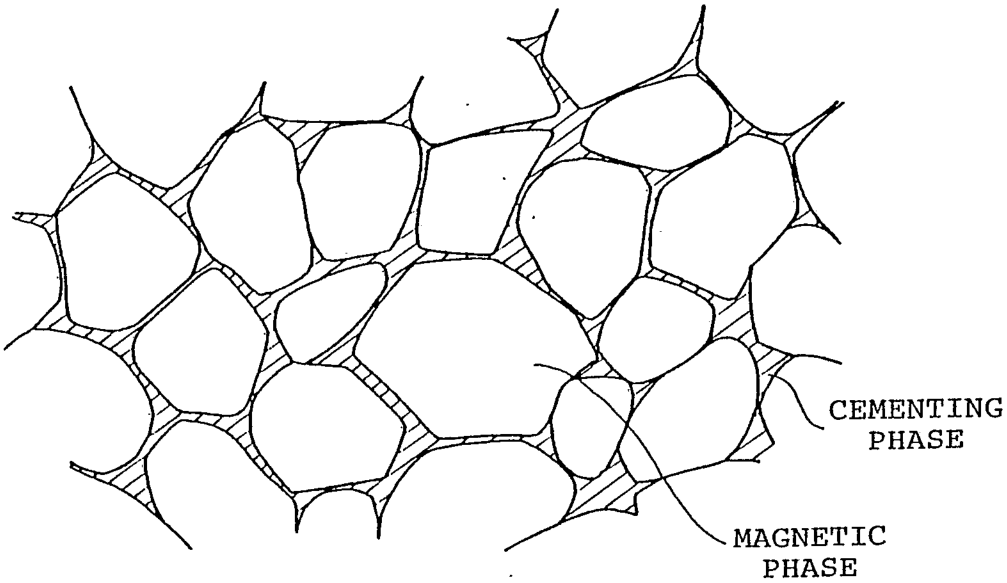

FIG. 1

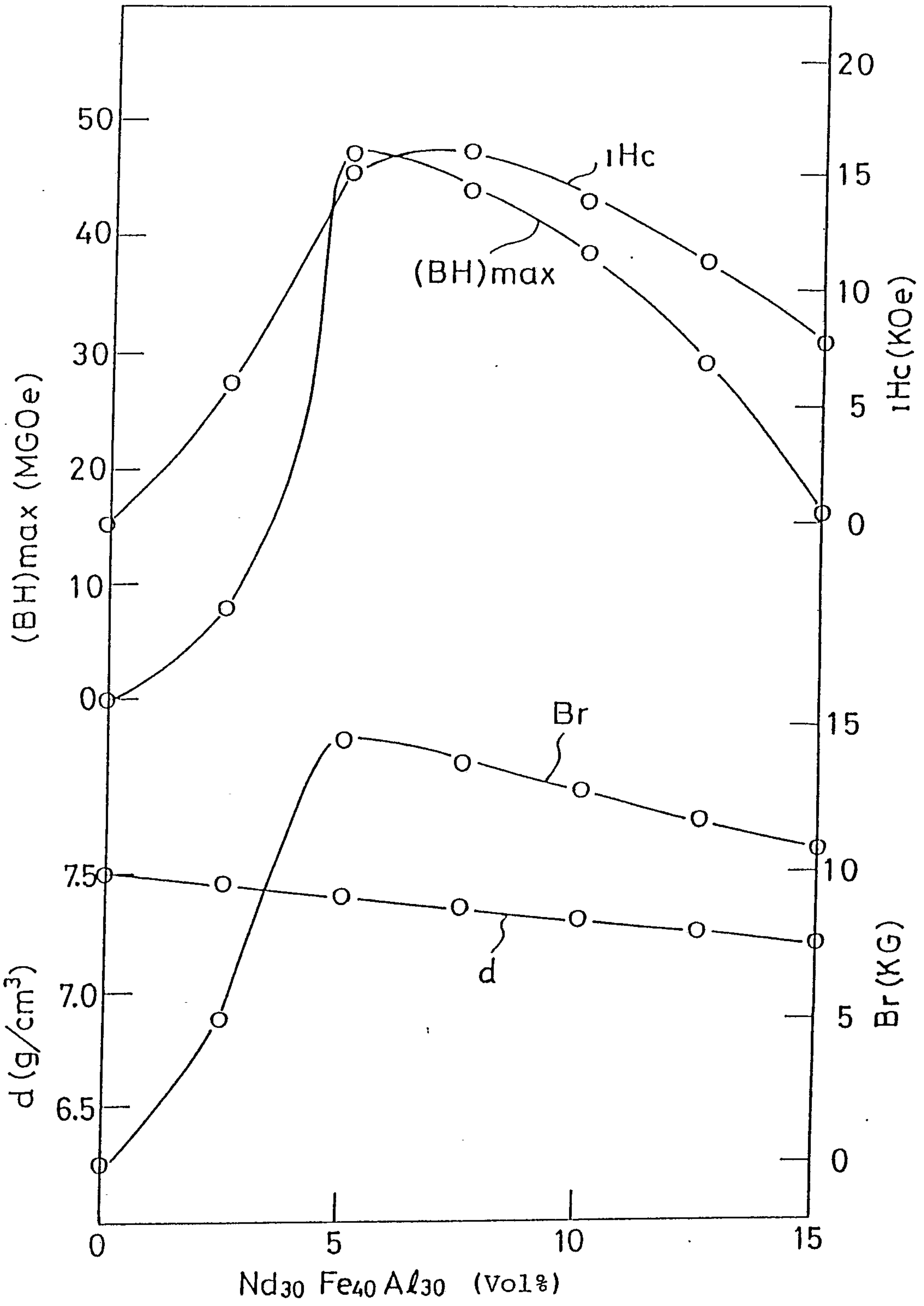
50
40
30
20
10
0
(BH)max (MGOe)
20
15
10
5
0
IHc (KOe)
IHc
(BH)max
Br
15
10
5
0
Br (KG)
7.5
7.0
6.5
d (g/cm³)
d
0
5
10
15
Nd30 Fe40 Aℓ30 (Vol%)

FIG. 2

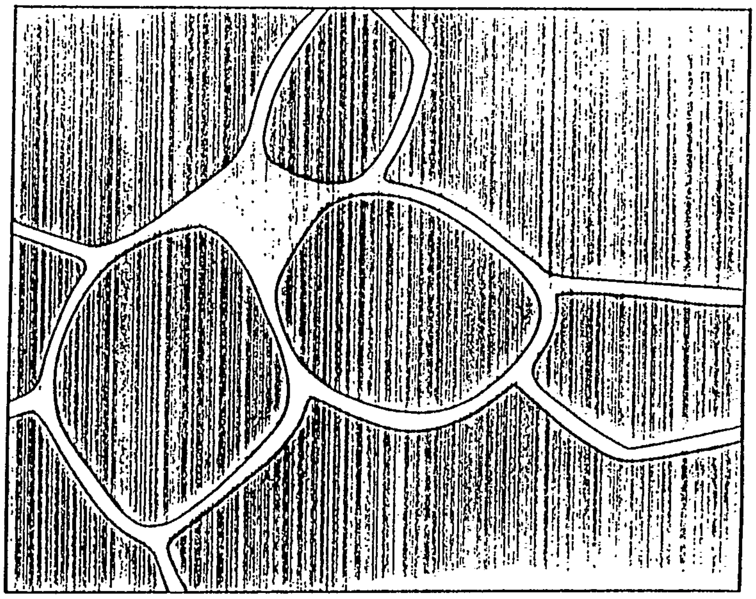

FIG. 3

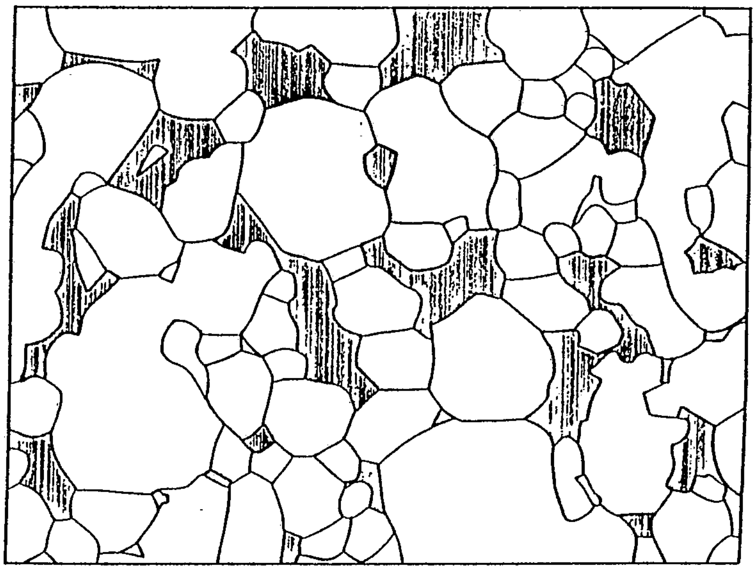

FIG. 4

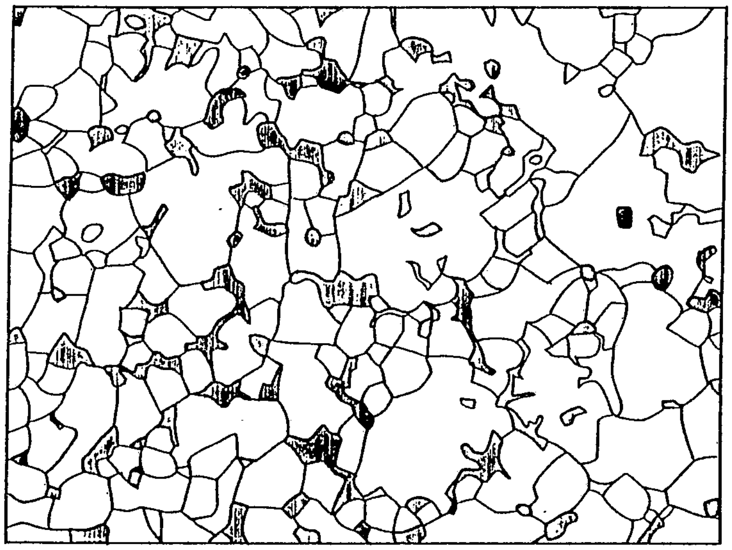

FIG. 5

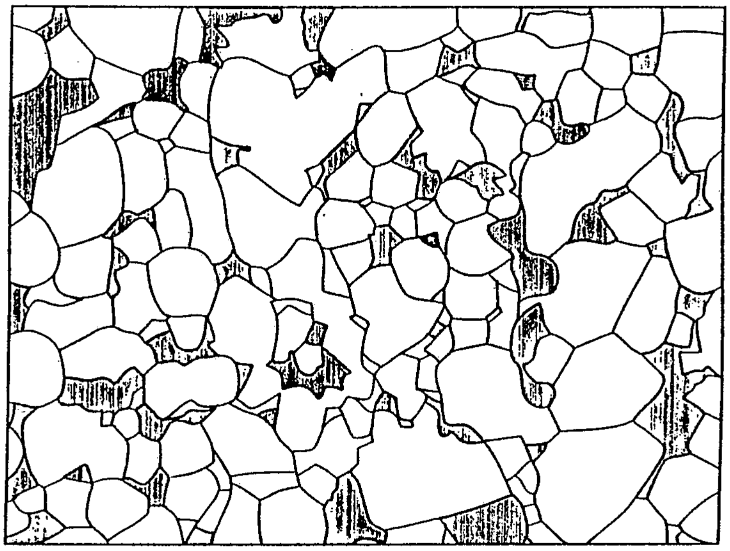

FIG.6

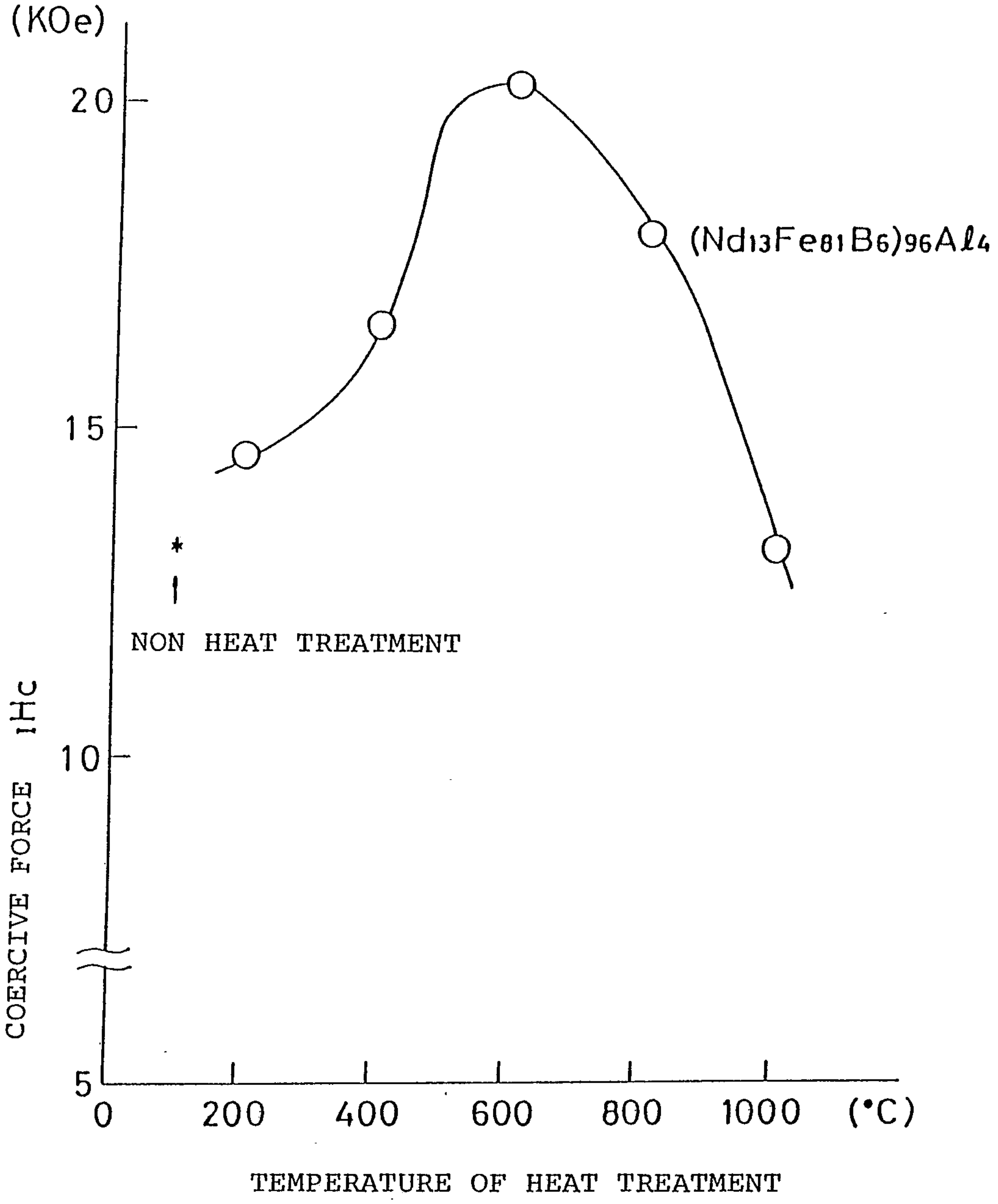
(KOe)
20
15
10
5
COERCIVE FORCE iHc
*
NON HEAT TREATMENT
(Nd13Fe81B6)96Al4
0
200
400
600
800
1000
(°C)
TEMPERATURE OF HEAT TREATMENT

FIG.7

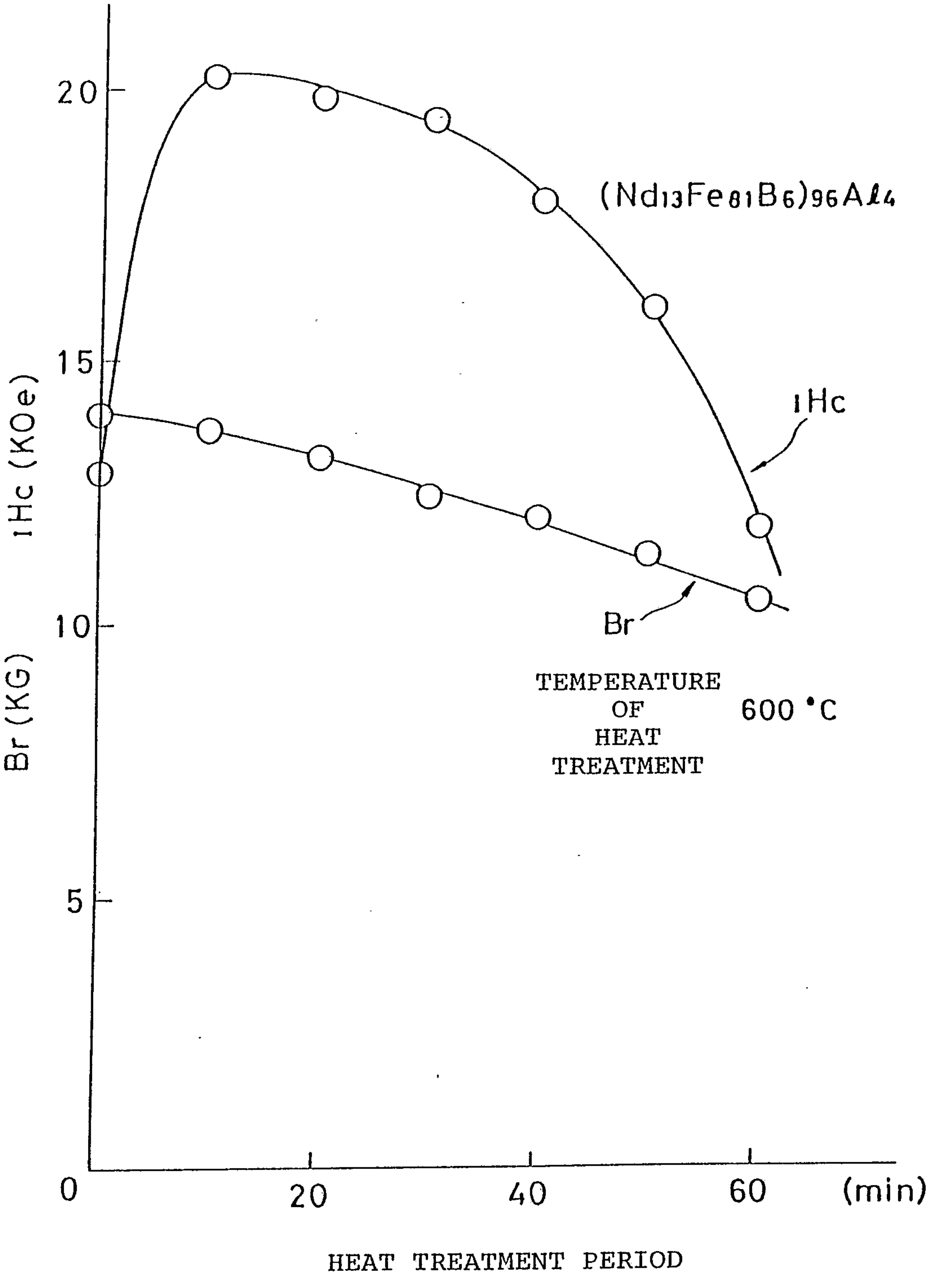
20
15
10
5
0
iHc (KOe)
Br (KG)
(Nd13Fe81B6)96Al4
iHc
Br
TEMPERATURE OF HEAT TREATMENT 600 °C
20
40
60
(min)
HEAT TREATMENT PERIOD

FIG.8

European Patent
Office

EUROPEAN SEARCH REPORT

0 249 973

Application number

EP 87108724.3

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
D,A	IEEE TRANSACTIONS ON MAGNETICS, vol. MAG-8, no. 3, September 1972, Kyoto, Japan KARL J. STRNAT "The Hard-Magnetic Properties of Rare Earth-Transition Metal Alloys" pages 511-516 * Totality * --	1	H 01 F 1/06
D,A	APPLIED PHYSICS LETTERS, vol. 39, no. 10, November 15, 1981, Washington D.C. N.C. KOON, B.N. DAS "Magnetic properties of amorphous and crystallized $(Fe_{0,82}B_{0,18})_{0,9}Tb_{0,5}La_{0,05}$" pages 840-842 * Totality * --	1	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
D,A	JOURNAL OF APPLIED PHYSICS, vol. 55, no. 6, part IIA, March 15, 1984, Michigan J.J. CROAT et al. "Pr-Fe and Nd-Fe-based materials: A new class of high-performance permanent magnets" pages 2078-2082 * Totality * --	1	H 01 F 1/00
A	EP - A2 - 0 166 597 (MITSUI TOATSU) * Abstract; claims 1-8 * ----	1	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	30-09-1987	VAKIL

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
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