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

(11) EP 0 522 177 B2

(12) **NEW EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention
of the opposition decision:
30.07.2003 Bulletin 2003/31

(45) Mention of the grant of the patent:
12.07.1995 Bulletin 1995/28

(21) Application number: **92903728.1**

(22) Date of filing: **28.01.1992**

(51) Int Cl.^{7}: **H01F 1/053**

(86) International application number:
PCT/JP92/00073

(87) International publication number:
WO 92/013353 (06.08.1992 Gazette 1992/21)

(54) Anisotropic Rare Earth Magnet

Anisotroper Seltenerd-Magnet

Aimant ayant des propriétés anisotropiques à base de terres rares

(84) Designated Contracting States:
DE FR GB IT NL

(30) Priority:
28.01.1991 JP 6082891
30.01.1991 JP 6083391
31.01.1991 JP 6083791
01.02.1991 JP 6086091

(43) Date of publication of application:
13.01.1993 Bulletin 1993/02

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Printed by Jouve, 75001 PARIS (FR)

Description

FIELD OF THE INVENTION

[0001] The present invention relates to a R (where R represents at least one type of rare earth element including Y) -Fe-Co-B system anisotropic magnet possessing superior magnetic anisotropy and a small coercive force temperature coefficient, and relates more particularly to an anisotropic magnet comprising a hot press molded body or a hot isostatic press molded body.

PRIOR ART

[0002] In Japanese Patent Application, First Publication, Laid-Open no. Hei 1-132106, a R-Fe-B system permanent magnetic powder obtained by means of the hydrogen treatment of a R-Fe-B system mother alloy, and a R-Fe-Co-B system permanent magnetic powder obtained by the hydrogen treatment of a R-Fe-Co-B System mother alloy, are disclosed.

[0003] The above R-Fe-B system permanent magnetic powder utilizes R-Fe-B system mother alloy as a raw material for the main phase of a $R_2Fe_{14}B$ type intermetallic compound phase (hereinbelow termed the "$R_2Fe_{14}B$ type phase") which is a ferromagnetic phase; after the heat treatment of this mother alloy raw material in a H_2 atmosphere within a specified temperature range and the fostering of a phase-changed state in each of the RH_x, Fe_2B, and the remaining Fe phases, the H_2 is removed from the raw material by means of an H_2-desorption process, and the $R_2Fe_{14}B$ type phase, which is the ferromagnetic phase, is recreated; the $R_2Fe_{14}B$ system permanent magnetic powder obtained as a result has an aggregate structure such that the main phase thereof is an extremely fine $R_2Fe_{14}B$ type phase having a recrystallized structure and an average grain diameter of 0.05 - 3 μm.

[0004] Furthermore, in the same manner, the above R-Fe-Co-B system permanent magnetic powder uses R-Fe-Co-B system mother alloy having as a main phase thereof $R_2(Fe, Co)_{14}B$ type intermetallic compound (hereinbelow termed $R_2(Fe, Co)_{14}B$ type phase), which is a ferromagnetic phase, as a raw material thereof, and this is processed in a manner identical to the case of the above R-Fe-B system; this has an aggregate structure having as a main phase thereof an extremely fine $R_2(Fe, Co)_{14}B$ type phase with a recrystallized structure and an average grain diameter of 0.05 - 3 μm.

[0005] EP-A-0 274 034 discloses magnetically anisotropic magnetic powders including R-Fe-B-Ga alloys which may optionally contain further elements such as V, Si and Al. The process of manufacturing these powders comprises inter alia the steps of hot pressing and plastic working of the material.

[0006] EP-A-0 304 054 discloses a process for producing a rare earth-iron-boron alloy magnet powder comprising the steps of:

(a) preparing a rare earth-iron-boron alloy material;
(b) subsequently occluding hydrogen into said alloy material at a temperature of 500 to 1000°C in an atmosphere of hydrogen gas optionally mixed with an inert gas
(c) subsequently subjecting said alloy material to dehydrogenation at a temperature of 500°C to 1000°C until the pressure of hydrogen in said atmosphere is decreased to no greater than 1×10^{-1} torr ; and
(d) subsequently cooling said alloy material.

The so-produced powder is then blended with a resin and compression moulded in a magnetic field followed by a heat treatment to solidify the resin to produce a bonded magnet.

[0007] The above R-Fe-B system and R-Fe-Co-B system permanent magnetic powders are unable to achieve sufficient magnetic anisotropy simply as a result of being formed as hot press molded bodies, so that, as disclosed in Japanese Patent Application, First Publication, Laid-Open No. Hei 2-39503, by carrying out a hot rolling process such as hot rolling or the like on the above hot press molded bodies and thus creating a rolled structure, the C axes of the crystal grains of the $R_2Fe_{14}B$ phase or the $R_2(Fe, Co)_{14}B$ type phase are oriented, and the magnetic anisotropy thereof is increased.

[0008] However, R-Fe-B system and R-Fe-Co-B system rolled magnets obtained by the further hot rolling of a hot press molded body possess superior magnetic anisotropy; but in comparison with magnets which are produced by the hot pressing of the above R-Fe-B system and R-Fe-Co-B system permanent magnetic powders obtained by means of hydrogen treatment, the temperature coefficient of the coercive force increases in an undesirable manner, and in the case in which such a rolled magnet is incorporated in a motor or the like, the performance of such a motor or the like varies based on temperature, and there is a problem in that the stability thereof is lacking.

[0009] Furthermore, in the above R-Fe-B system and R-Fe-Co-B system rolled magnets, positional variation in the degree of working causes variations in magnetic anisotropy, so that in order to prevent this, it is impossible to avoid an increase in the complexity of the hot plastic working processes.

[0010] Believing that the above increase in the temperature coefficient of the coercive force was caused as a result of the hot rolling of a hot press molded body, and based on the conviction that if a magnet having superior magnetic anisotropy could be obtained without the use of hot rolling, this increase in the temperature coefficient of the coercive force would not occur, the present inventors have conducted research, and have obtained the R-Fe-B system and R-Fe-Co-B system anisotropic magnets of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0011] The first anisotropic magnet prepared in accordance with the present invention is prepared by a first method as defined by Claim 1.

[0012] This first anisotropic magnet has a temperature coefficient of the coercive force which is small, and in comparison with the conventional rolled magnets, has almost no localized variations in magnetic anisotropy and also possesses superior resistance to corrosion. Furthermore, as the magnet has crystallized grain aggregate structure, it has superior magnetic anisotropy and a high coercive force in the vicinity of a $R_2(Fe, Co)_{14}B$ type compound composition, that is to say, in the vicinity of a $R_{11.8}(Fe, Co)_{bal}B_{5.9}$ (atomic %) composition.

[0013] It is acceptable to add one or a plurality of Al, V, and Si in a total amount of 0.01 - 2.0 atomic % to the composition of this first anisotropic magnet. In such a case, the maximum energy product will be further increased.

[0014] In manufacturing this first R-Fe-Co-B system anisotropic magnet, first, a R-Fe-Co-B system mother alloy having a fixed component composition possessing Ga, Zr, and Hf, or a R-Fe-Co-B system mother alloy having a fixed component composition in which Al, V, and Si are added to the above alloy, is manufactured.

[0015] Next, this R-Fe-Co-B system mother alloy is heated in an atmosphere of hydrogen gas, is subjected to heat treatment at a temperature of 500 - 1000°C in an atmosphere of hydrogen gas or a mixed atmosphere of hydrogen gas and an inert gas, and then hydrogen removal processing is carried out at a temperature of 500 - 1000°C so as to produce a vacuum atmosphere having a hydrogen gas pressure of less than 1 Torr or an inert gas atmosphere in which the partial pressure of hydrogen gas is less than 1 Torr, and by cooling this, a R-Fe-Co-B system permanent magnetic powder is obtained.

[0016] By means of the addition of a procedure for homogenizing processing at a temperature of 600 - 1200°C prior to the conducting of the above heat treatment of the R-Fe-Co-B system mother alloy above, and by means of the addition of a procedure for heat treatment at a temperature of 300 - 1000°C after the hydrogen desorped processing above, it is possible to create a R-Fe-Co-B system permanent magnetic powder having more superior magnetic anisotropy and resistance to corrosion.

[0017] The structure of the R-Fe-Co-B system permanent magnetic powder produced in the above manner comprises a recrystallized aggregate structure in which $R_2(Fe, Co)_{14}B$ type intermetallic compound phase recrystallized grains, which are free of impurities or strains within the grains or at the grain boundaries, are aggregated. The average recrystallized grain diameter of the recrystallized grains comprising this recrystallized aggregate structure is sufficiently within a range of 0.05 - 20 μm; however, a range of 0.05 - 3 μm, which is dose to the dimensions of a single magnetic domain grain diameter (approximately 0.3 μm), is more preferable.

[0018] It is preferable that the individual recrystallized grains having the above dimensions have a form such that the value of the ratio b/a of the smallest grain diameter a and the largest grain diameter b be less than 2; it is necessary that recrystallized grains having this form be present in an amount of more than 50 volume % of the total recrystallized grains comprising the structure of the individual powders. By means of setting the form of the recrystallised grains so that the ratio b/a of the smallest grain diameter a and the largest grain diameter b have a value of less than 2, the coercive force of the R-Fe-Co-B system permanent magnetic powder is improved, and the coercive force temperature coefficient αiHc in the temperature range of 25°C - 100°C becomes smaller than 0.6%/°C.

[0019] Furthermore, as the recrystallized structure of the R-Fe-Co-B system permanent magnetic powder produced in this manner has a recrystallised aggregate structure comprising materially only $R_2(Fe, Co)_{14}B$ type intermetallic compound phase in which a grain boundary phase is almost nonexistent, it is possible to raise the magnetization values of only the portion having no grain boundary phase, corrosion proceeding along the grain boundary phase is halted, and furthermore, as stress deformation resulting from thermoplastic processes does not exists, the likelihood of stress corrosion is small, and the resistance to corrosion thus increases.

[0020] Next, the R-Fe-Co-B system permanent magnetic powder above is pressed to a green compact in a magnetic field, and by subjecting this green compact to hot pressing or a HIP process at a temperature of 600°C - 900°C, it is possible to produce a R-Fe-Co-B system anisotropic magnet which preserves the superior characteristics of the R-Fe-Co-B system permanent magnetic powder above. Furthermore, by conducting heat processing at 300°C - 1000°C where necessary, it is possible to increase the coercive force.

[0021] When the green compact above is sintered by a conventional method, as the sintering temperature is normally high, the fine recrystallized grains of the R-Fe-Co-B system permanent magnetic powder grow into large crystallized grains, and as the magnetic characteristics, and particularly the coercive force, worsen, this is not preferable. Furthermore, as the imparting of magnetic anisotropy is conducted in a magnetic field, it is not necessary to conduct a thermoplastic process after hot pressing or the HIP process.

[0022] The reasons for the limitation of the component composition, average crystallized grain diameter, and crystallized grain form of the first R-Fe-Co-B system anisotropic magnet are as follows.

(a) R
R exhibits one or a plurality of Nd, Pr, Tb, Dy, La, Ce, Ho, Er, Eu, Sm, Gd, Tm, Yb, Lu, and Y; in general, Nd is used as a main element, and to this are added other rare earth elements, and in particular, Tb, Dy, and Pr have the effect of increasing the coercive force iHc.

(b) B
It is possible to substitute one or a plurality of C, N, O, P, and F for a portion of B; this is also the case with the second anisotropic magnet described hereinafter.

(c) Ga, Zr, and Hf
Ga, Zr, and Hf have the function of increasing the coercive force and also stably imparting superior magnetic anisotropy and resistance to corrosion; however, if one or a plurality of Ga, Zr, and Hf are contained in a total amount of less than 0.001 atomic %, the desired effects cannot be obtained, while on the other hand, when the total amount contained is greater than 5.0 atomic %, the magnetic characteristics worsen. Accordingly, the total amount contained of 1 or a plurality of Ga, Zr, and Hf is set to a level of 0.001 - 5.0 atomic %.

(d) Al, V, and Si
Where necessary, Al, V, and Si may be added as components of the R-Fe-Co-B system anisotropic magnet. These exhibit the effect of increasing the coercive force; however, if one or a plurality of Al, V, and Si are contained in a total amount of less than 0.01 atomic %, the desired effects cannot be obtained, while on the other hand, when this amount exceeds 2.0 atomic %, the magnetic characteristics worsen. Accordingly, it is preferable that one or a plurality of Al, V, and Si be contained in a total amount of 0.01 - 2.0 atomic %.

(e) Average Crystallized Grain Diameter and Form Thereof
If the average crystallized grain diameter of the crystallized grains comprising the structure of the anisotropic magnet is smaller than 0.05 μm, magnetization becomes a problem, so that this is not desirable, while on the other hand, at a value of more than 20 μm, the coercive force and the angularity of the hysteresis loop is reduced, and furthermore, the temperature coefficient of the coercive force increases, so that this is also not desirable. Accordingly, the average crystallized grain diameter is set to a value of 0.05 - 20 μm. It is more preferable that the average crystallized grain diameter be within the range of 0.05 - 3 μm, which is close to the dimensions of the simple magnetic domain grain diameter (0.3 μm). It is preferable that the individual crystallized grains have a value of less than 2 for the ratio b/a, the ratio of the smallest grain diameter a and the largest grain diameter b; it is necessary that crystallized grains having such a form be present in an amount of greater than 50 volume % of the total crystallized grains. By means of so setting the form of the crystallized grains so that a value of less than 2 is obtained for the ratio b/a between the smallest grain diameter a and the largest grain diameter b, the coercive force of the R-Fe-Co-B system anisotropic magnet is improved, the resistance to corrosion increases, and the temperature coefficient of the coercive force is reduced. Accordingly, the value of b/a of the individual crystallized grains is set to less than 2.

[0023] With respect to the amount of Co contained, by adding Co to the composition of the anisotropic magnet, the coercive force and magnetic temperature characteristics (for example, the Curie point) of the anisotropic magnet are improved, and moreover, the effect of an increase in the resistance to corrosion is obtained; however, when the amount contained thereof is less than 0.1 atomic %, these effects cannot be obtained, while on the other hand, when the amount exceeds 50 atomic %, the magnetic characteristics worsen, so that this is not preferable. Accordingly, the amount of Co contained is set to a range of 0.1 - 50 atomic %. When the amount of Co contained is in a range of 0.1 - 20 atomic %, the coercive force increases to the greatest extent, so that it is most preferable to set the amount of Co contained to 0.1 - 20 atomic %.

[0024] Furthermore, the reasons for the limitation of the preferable range of amounts of Al, V, and Si, contained are the same as in the case of the second anisotropic magnet which are discussed below.

[0025] The second anisotropic magnet in accordance with the present invention is a R-Fe-Co-B system anisotropic magnet as defined by Claim 3.

[0026] As in the case of the first anisotropic magnet above, this second anisotropic magnet has a small coercive force temperature coefficient, has almost no localized variations in magnetic anisotropy in comparison with conventional rolled magnets, has superior corrosion resistance, and as this magnet possesses a crystallized grain aggregate structure, it has superior magnetic anisotropy and a high coercive force even in the vicinity of a $R_2(Fe, Co)_{14}B$ type compound composition, that is to say, in the vicinity of a $R_{11.8}(Fe, Co)_{bal}B_{5.9}$ (atomic %) composition.

[0027] In order to produce this second anisotropic magnet, first, a R-Fe-Co-B system mother alloy having a fixed component composition containing one or a plurality of Ti, V, Nb, Ta, Al, and Si is subjected to melt casting, and using this as a raw material, it is acceptable to conduct processing which is identical to that in the case of the first anisotropic magnet above.

[0028] The reasons for the limitation of R, B, Co, the average crystallized grain diameter, and the crystallized grain form in the component composition of the anisotropic magnet of the present invention as given above are the same as in the case of the first anisotropic magnet which was previously discussed.

[0029] With respect to Ti, V, Nb, Ta, Al, and Si, by means of adding one or a plurality of these elements to the components of the R-Fe-B system anisotropic magnet, the effects of an increase in the coercive force and the stable imparting of superior magnetic anisotropy and resistance to corrosion can be obtained; however, when the total amount of these elements which is contained is less than 0.001 atomic %, the desired effects cannot be obtained, while on the other hand, when this amount exceeds 5.0 atomic %, the magnetic characteristics worsen. Accordingly, the total amount of one or a plurality of Ti, V, Nb, Ta, Al, and Si which is contained is set to a value in the range of 0.001 - 5.0 atomic %.

[0030] Even if this second anisotropic magnet contains at least one of Ni, Cu, Zn, Ga, Ge, Zr, Mo, Hf, and W in an amount of 0.001 - 5.0 atomic %, it possesses superior magnetic anisotropy and resistance to corrosion.

EXAMPLES

[0031] Next, the first and second anisotropic magnets in accordance with the present invention were produced in the following manner, and the characteristics thereof were determined.

(Examples of the First Anisotropic Magnet)

[0032] A first anisotropic magnet in accordance with the present invention was produced in the manner described hereinafter, and the characteristics thereof were determined.

[0033] Ingots of various R-Fe-Co-B system alloys containing Co and one or a plurality of Ga, Zr, and Hf, and ingots of R-Fe-Co-B system alloys containing no Ga, Zr, or Hf which were obtained by means of plasma melting and casting, were prepared, these alloy ingots were subjected to homogenizing processing in an argon gas atmosphere under conditions such that the temperature thereof was 1120°C, and the processing time was 40 hours, and thereafter, these homogenizing processed ingots were crushed to a fineness of 20 mm to form a raw material alloy.

[0034] The temperature of this raw material alloy was raised from room temperature to a temperature of 830°C in a hydrogen atmosphere at a pressure of 101 kPa (1 atmosphere), heat treatment was conducted in this hydrogen atmosphere at a temperature of 830°C for a period of 4 hours, and then, hydrogen was desorped at a temperature of 830°C so as to produce a vacuum degree of less than 13 Pa (1×10^{-1} Torr), and directly thereafter, argon gas was introduced and rapid cooling conducted.

[0035] After the conclusion of the above hydrogen treatment, the ingots were slightly crushed in mortars, and various R-Fe-Co-B system permanent magnetic powders having a mean particle size of 50 μm were obtained.

[0036] These R-Fe-Co-B system permanent magnetic powders were press formed in a magnetic field so as to form green compacts, and these green compacts were subjected to hot pressing under conditions such that the temperature was 700°C and the pressure was 1.5 Ton/cm^2. At this time, arrangement and hot pressing were conducted in such a manner that the orientation direction was identical with the press direction at the time of hot pressing.

[0037] By means of further subjecting the various molded bodies to heat processing at a temperature of 620°C and for a period of 2 hours, the anisotropic magnets 79 - 109 of the present invention and the comparative anisotropic magnets 30 - 39 shown in Tables 20 - 23 were produced. The densities of these anisotropic magnets were sufficiently accurate, being in a range of 7.5 to 7.6 g/cm^3.

[0038] On the other hand, a R-Fe-Co-B system permanent magnetic powder produced from an ingot of an alloy containing no Ga, Zr, or Hf was placed in a copper can in a vacuum, this was heated to a temperature of 700°C and rolling was conducted a number of times so that the rolling ratio reached 80%, and the conventional anisotropic magnet 3 shown in Table 23 was obtained.

[0039] The various structures of the anisotropic magnets 79 - 109 of the present invention, the comparative anisotropic magnets 30 - 39, and the conventional anisotropic magnet 3 having the component compositions shown in Tables 20 - 22 were observed by means of a scanning electron microscope, and the average crystallized grain diameter, the amount of crystallized grains present having a form in which the ratio of the largest grain diameter / smallest grain diameter had a value of less than 2, the coercive force temperature coefficient αiHc and the magnetic characteristics were determined. The values obtained thereby are shown in Tables 24 - 27. The method of calculation of the coercive force temperature coefficient αiHc is as given above.

[0040] It is clear from the results shown in Tables 20 - 27 that the anisotropic magnets 79 - 109 In accordance with the present invention which contain one or a plurality of Ga, Zr, and Hf have superior magnetic characteristics, and in particular, have superior maximum energy product $(BH)_{max}$ and residual magnetic flux density Br, and furthermore, have superior anisotropy. Furthermore, in comparison with the conventional anisotropic magnet 3 obtained by means of rolling, the anisotropic magnet 79 - 109 of the present invention possess essentially identical magnetic characteristics; however, the coercive force temperature coefficient αiHc thereof is markedly smaller, having a value of approximately -0.5%/°C. In addition, in the case of the comparative anisotropic magnets 30 - 39, the compositions of which lie outside the ranges of the present invention, the magnetic characteristics and magnetic anisotropy worsen.

TABLE 20

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)										
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT				Fe
								Ga	Zr	Hf		
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	79	12.3	-	-	0.3	11.6	6.5	0.01	-	-	0.01	*
	80	12.0	-	-	0.2	11.6	6.5	0.5	-	-	0.5	*
	81	12.0	-	0.5	-	11.6	6.4	1.0	-	-	1.0	*
	82	12.0	-	0.3	-	11.6	6.2	5.0	-	-	5.0	*
	83	12.1	0.2	-	0.2	11.6	6.3	-	0.01	-	0.01	*
	84	12.2	0.2	-	0.2	11.6	6.3	-	0.1	-	0.1	*
	85	12.1	0.1	-	0.3	11.6	6.2	-	1.0	-	1.0	*
	86	12.1	0.2	-	0.2	11.6	6.3	-	5.0	-	5.0	*
	87	12.0	-	0.2	0.2	11.6	6.3	-	-	0.01	0.01	*
	88	12.2	-	0.2	0.2	11.6	6.4	-	-	0.1	0.1	*
	89	12.2		0.2	0.2	11.6	6.3	-	-	1.0	1.0	*
	90	12.0	-	0.2	0.3	11.6	6.2	-	-	5.0	5.0	*
	91	12.0	-	0.3	-	11.6	6.3	-	0.2	0.1	0.3	*
	92	12.3	-	0.3	-	11.6	6.3	-	2.0	2.0	4.0	*

* balance

TABLE 21

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)										
		Nd	Tb	Dy	Pr	Co	B	Ga	Zr	Hf	TOTAL AMOUNT	Fe
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	93	12.2	–	–	0.3	17.0	6.0	0.001	0.001	0.001	0.003	*
	94	12.5	–	–	–	17.4	6.0	0.5	0.1	–	0.6	*
	95	12.2	–	–	0.3	18.5	6.0	0.4	–	0.1	0.5	*
	96	10.0	–	–	–	5.0	8.0	1.0	–	–	1.0	*
	97	15.0	–	–	–	17.5	8.0	0.5	–	0.1	0.6	*
	98	20.0	–	–	–	17.6	7.0	1.0	–	0.1	1.1	*
	99	12.2	–	0.4	–	0.1	6.0	0.5	–	–	0.5	*
	100	12.4	–	0.3	–	5.2	6.0	0.5	–	–	0.5	*
	101	12.3	–	0.3	–	17.5	6.0	0.5	–	–	0.5	*
	102	12.4	–	0.2	–	30.0	6.0	0.5	–	–	0.5	*
	103	12.3	–	0.3	–	50.0	6.0	0.5	–	–	0.5	*

* balance

TABLE 22

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)										
		Nd	Tb	Dy	Pr	Co	B	Ga	Zr	Hf	TOTAL AMOUNT	Fe
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	104	16.0	–	–	–	11.2	3.0	1.0	–	0.1	1.1	*
	106	12.1	–	–	0.5	6.4	10.4	–	0.1	0.1	0.2	*
	106	14.0	–	–	–	11.0	20.0	–	0.1	–	0.1	*
	107	13.0	–	0.5	–	11.5	6.1	0.5	–	–	0.5	*
	108	13.0	–	0.5	–	11.5	6.1	0.5	–	–	0.5	*
	109	16.0	–	–	–	11.3	6.0	0.5	–	–	0.5	*

* balance

TABLE 23

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)										
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT				Fe
								Ga	Zr	Hf		
COMPARATIVE ANISOTROPIC MAGNETS	30	12.5	–	–	–	7.0	6.0	0.5	–	–	0.5	*
	31	16.0	–	–	–	7.0	8.0	0.5	–	–	0.5	*
	32	12.5	–	0.2	–	7.1	6.3	7.9※	–	–	7.9※	*
	33	12.3	–	0.3	–	7.2	6.2	–	7.0※	0.2	7.2※	*
	34	12.3	–	0.2	–	7.0	6.2	–	0.2	6.7※	6.9※	*
	35	9.0※	–	–	–	16.2	8.0	–	0.1	–	0.1	*
	36	25.0※	–	–	–	16.5	8.0	0.5	–	–	0.5	*
	37	13.0	–	–	–	55.1※	7.0	1.0	–	0.5	1.5	*
	38	16.0	–	–	–	11.2	2.0※	–	–	0.1	0.1	*
	39	14.0	–	–	–	11.2	21.7※	–	0.1	0.1	0.2	*
CONVENTIONAL ANISOTROPIC MAGnet 3		14.5	–	–	–	17.5	8.0	–	–	–	–	*

(※ INDICATES VALUES WHICH DO NOT FULFILL THE CONDITIONS OF THE PRESENT INVENTION)

* balance

TABLE 24

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μ m)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α I H c (% / °C)	MAGNETIC CHARACTERISTICS		
					B r (KG) 10^{-1}T	I H c (Koe) kA/m	B H max (MGOe) kJ/m^3
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	79	0.2	80	-0.50	11.3	1064 (13.3)	226.4 (28.3)
	80	0.3	90	-0.50	12.4	1152 (14.4)	283.2 (35.4)
	81	0.3	80	-0.49	12.6	1120 (14.0)	289.6 (36.2)
	82	1.0	80	-0.48	12.5	968 (12.1)	280 (35.0)
	83	0.3	90	-0.50	11.8	960 (12.0)	252 (31.5)
	84	0.2	9.0	-0.49	12.9	848 (10.6)	320.8 (40.1)
	85	0.3	100	-0.49	12.6	768 (9.6)	293.6 (36.7)
	86	0.05	90	-0.48	11.4	680 (8.5)	242.4 (30.3)
	87	0.4	80	-0.51	11.8	864 (10.8)	248 (31.0)
	88	0.3	80	-0.50	12.8	872 (10.9)	307.2 (38.4)
	89	0.2	80	-0.50	12.6	840 (10.5)	295.2 (36.9)
	90	0.1	90	-0.48	11.5	768 (9.6)	244 (30.5)
	91	0.2	90	-0.51	11.8	880 (11.0)	256.8 (32.1)
	92	0.05	90	-0.50	11.5	736 (9.2)	245.6 (30.7)

TABLE 25

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μ m)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α I H c (% / °C)	MAGNETIC CHARACTERISTICS		
					B r (KG) 10^{-1}T	I H c (Koe) kA/m	B H max (MGOe) kJ/m^3
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	93	0.5	90	-0.52	10.7	912 (11.4)	204.8 (25.6)
	94	0.2	90	-0.49	13.1	1120 (14.0)	324.8 (40.6)
	95	0.2	90	-0.49	13.0	1104 (13.8)	320 (40.0)
	96	0.1	80	-0.53	11.2	656 (8.2)	206.4 (25.8)
	97	0.3	100	-0.51	12.4	1304 (16.3)	276 (34.5)
	98	0.5	100	-0.53	11.2	920 (11.5)	213.6 (26.7)
	99	0.5	90	-0.52	11.6	776 (9.7)	260.8 (32.6)
	100	0.3	90	-0.52	12.1	968 (12.1)	272 (34.0)
	101	0.3	90	-0.51	13.0	960 (12.0)	316 (39.5)
	102	0.1	90	-0.48	12.2	864 (10.8)	231.2 (28.9)
	103	0.5	90	-0.48	10.9	696 (8.7)	208.8 (26.1)

TABLE 26

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μm)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α_{IHc} (%/°C)	MAGNETIC CHARACTERISTICS		
					Br (KG) 10^{-1}T	IHc (Koe) kA/m	BHmax (MGOe) kJ/m^3
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	104	0.3	90	-0.52	11.2	664 (8.3)	180.8 (22.6)
	105	0.3	80	-0.49	11.4	760 (9.5)	201.6 (25.2)
	106	0.4	90	-0.51	10.7	496 (6.2)	168 (21.0)
	107	0.8	60	-0.53	12.4	1064 (13.3)	252.8 (31.6)
	108	1.0	50	-0.53	12.6	992 (12.4)	270.4 (33.8)
	109	3.0	85	-0.53	11.5	848 (10.6)	220.8 (27.6)

TABLE 27

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μ m)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α I H c (% / °C)	MAGNETIC CHARACTERISTICS		
					B r (KG) 10^{-1}T	I H c (Koe) kA/m	B H max (MGOe) kJ/m^3
COMPARATIVE ANISOTROPIC MAGNETS	30	0.01※	90	-0.40	8.5	240 (3.0)	43.2 (5.4)
	31	25※	70	-0.74	5.9	200 (2.5)	(< 5) < 40
	32	0.5	80	-0.53	9.2	640 (8.0)	84.8 (10.6)
	33	0.3	90	-0.54	9.9	312 (3.9)	80.8 (10.1)
	34	0.3	80	-0.54	9.5	328 (4.1)	76.8 (9.6)
	35	1.0	90	—	4.5	48 (0.6)	(< 5) < 40
	36	5.0	80	—	8.5	168 (2.1)	(< 5) < 40
	37	0.3	70	-0.53	6.5	440 (5.5)	(< 5) < 40
	38	1.0	70	—	3.2	72 (0.9)	(< 5) < 40
	39	1.0	90	—	3.4	80 (1.0)	(< 5) < 40
CONVENTIONAL ANISOTROPIC MAGNET 3		0.8	40※	-0.70	11.9	108 (13.5)	249.6 (31.2)

(※INDICATES VALUES WHICH DO NOT FULFILL THE CONDITIONS OF THE PRESENT INVENTION)

(Other Examples of the First Anisotropic Magnet)

[0041] Next, ingots of various alloys having component compositions containing one or a plurality of Al, V, and Si in addition to a R-Fe-Co-B system alloy containing one or a plurality of Ga, Zr, and Hf obtained by means of high frequency induction melting and casting, were prepared; these ingots were processed by a method identical to that of the anisotropic magnets 79 - 109 and the comparative anisotropic magnets 30 - 39 above, and R-Fe-Co-B system permanent magnetic powders having a mean particle size of 40 µm were produced. These permanent magnetic powders were press formed in the presence or absence of a magnetic field to form green compacts, these green compacts were subjected to hot isostatic pressing under conditions such that the temperature thereof was 710°C, and the pressure thereof was 1.7 Ton/cm^2, and the anisotropic magnets 110 - 119 of the present invention and the comparative anisotropic magnets 40 - 42 comprising the component compositions shown in Table 28 were obtained.

[0042] The average crystallized grain diameter, the amount (volume %) of crystallized grains present for which the value of the ratio of the largest grain diameter / smallest grain diameter was less than 2, the coercive force temperature coefficient αiHc and the magnetic characteristics of these anisotropic magnets were determined in a manner identical to that above. The results thereof are shown in Table 29.

[0043] As can be seen from the results of Tables 28 and 29, by means of adding 0.1 - 2.0 atomic % of one or a plurality of Al, V, and Si to 0.01 -5.0 atomic % of 1 or a plurality of Ga, Zr, and Hf, the maximum energy product is further increased. Furthermore, it is clear that the crystallized grain diameter and the form of the crystallized grains have a large effect in the reduction of the coercive force temperature coefficient.

TABLE 28

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)											
		N d	C o	B	TOTAL AMOUNT				TOTAL AMOUNT				F e
					G a	Z r	H f		A l	V	S i		
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	110	12.5	17.4	6.0	1.0	—	—	1.0	0.3	—	—	0.3	*
	111	12.5	17.5	6.0	1.0	—	—	1.0	0.1	0.1	—	0.2	*
	112	12.4	17.4	5.9	1.0	—	—	1.0	—	—	0.5	0.5	*
	113	12.4	17.4	6.0	—	0.1	—	0.1	0.5	—	—	0.5	*
	114	12.3	17.4	6.0	—	0.1	—	0.1	—	0.3	—	0.3	*
	115	12.4	17.4	6.0	—	0.1	—	0.1	—	—	0.5	0.5	*
	116	12.4	11.6	6.0	—	—	0.1	0.1	1.0	—	—	1.0	*
	117	12.5	11.6	6.0	—	—	0.1	0.1	—	0.2	1.2	1.4	*
	118	12.5	11.6	6.0	—	—	0.1	0.1	1.0	—	1.0	2.0	*
	119	12.5	11.6	6.0	0.5	0.1	—	0.6	0.05	0.02	0.01	0.08	*
COMPARATIVE ANISOTROPIC MAGNETS	40	12.5	17.5	6.0	1.0	—	—	1.0	3.0※	—	—	3.0※	*
	41	12.4	17.4	6.0	1.0	—	—	1.0	—	3.0※	—	3.0※	*
	42	12.5	17.5	6.0	1.0	—	—	1.0	—	—	3.0※	3.0※	*

(※ INDICATES VALUES WHICH DO NOT FULFILL THE CONDITIONS OF THE PRESENT INVENTION)

* balance

TABLE 29

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μ m)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α iHc (% / °C)	MAGNETIC CHARACTERISTICS		
					Br (KG) 10^{-1}T	1Hc (Koe) kA/m	BHmax (MGOe) kJ/m^3
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	110	0.3	80	-0.50	12.8	1152 (14.4)	309.6 (38.7)
	111	0.3	90	-0.50	12.9	1096 (13.7)	316 (39.5)
	112	0.2	80	-0.51	12.9	1184 (14.8)	316.8 (39.6)
	113	0.5	80	-0.48	13.3	984 (12.3)	338.4 (42.3)
	114	0.1	100	-0.49	13.3	808 (10.1)	37.6 (42.2)
	115	0.3	80	-0.49	13.5	1024 (12.8)	350.4 (43.8)
	116	0.3	90	-0.49	13.2	1040 (13.0)	336 (42.0)
	117	0.2	90	-0.50	13.3	856 (10.7)	337.6 (42.2)
	118	0.3	90	-0.49	13.5	1032 (12.9)	348 (43.5)
	119	0.3	90	-0.50	13.2	1152 (14.4)	330.4 (41.3)
COMPARATIVE ANISOTROPIC MAGNETS	40	0.5	70	-0.53	11.1	976 (12.2)	186.4 (23.3)
	41	0.8	80	-0.55	11.2	768 (9.6)	156 (19.5)
	42	0.5	70	-0.53	10.9	904 (11.3)	136 (17.0)

(Example of a Second Anisotropic Magnet)

[0044] Next, a second anisotropic magnet in accordance with the present invention was produced in the manner detailed hereinbelow, and the characteristics thereof were determined.

[0045] Ingots of a R-Fe-Co-B system alloy containing one or a plurality of Ti, V, Nb, Ta, Al, and Si, and ingots of a R-Fe-Co-B system alloy which contained no Ti, V, Nb, Ta, Al, or Si, which were obtained by means of plasma melting and casting, were subjected to homogenizing processing in an atmosphere of argon gas under conditions such that the temperature thereof was 1130°C, and the processing time thereof was 20 hours, and thereafter, these homogenizing processed ingots were crushed to a fineness of approximately 15 mm to form raw material alloys.

[0046] The temperature of these raw material alloys was raised from room temperature to 830°C in an atmosphere of hydrogen gas at a pressure of 101 kPa (1 atmosphere), heat treatment was carried out in an atmosphere of hydrogen gas at a temperature of 830°C for a period of 1 hour, hydrogen was desorped at a temperature of 830°C in order to produce a vacuum degree of less than 13 Pa (1 X 10^{-1} Torr), and immediately thereafter, argon gas was introduced and rapid cooling conducted. After the conclusion of this hydrogen treatment, heat processing was conducted in a vacuum at a temperature of 630°C for a period of 2 hours. The raw material alloys thus obtained were slightly crushed in mortars, and magnetic powders having a mean particle size of 40 μm were obtained.

[0047] These magnetic powders were press formed in a 25 KOe magnetic field to produce green compacts, and each green compact was subjected to hot pressing under conditions such that the temperature thereof was 720°C and the pressure thereof was 1.5 t/cm^2, or was subjected to a HIP process under conditions such that the temperature thereof was 710°C and the pressure thereof was 1.5 t/cm^2, and furthermore, each melted body was then subjected to heat processing at a temperature of 620°C for a period of 2 hours. The green compacts which were formed in a magnetic field were arranged and hot pressed in a such a manner that the orientation direction was identical with the pressing direction of the hot pressing.

[0048] Among the anisotropic magnets 120 - 164 of the present Invention and the comparative anisotropic magnets 43 - 56 which were produced in the above manner, anisotropic magnets 120 - 144 of the present invention and comparative anisotropic magnets 43 - 49 were produced by means of hot pressing, while anisotropic magnets 145 - 164 of the present invention and comparative anisotropic magnets 50 - 56 were produced by means of an HIP method. The density thereof was in all cases sufficiently accurate, being within a range of 7.5 - 7.6 g/cm^3.

[0049] Furthermore, for the purposes of comparison, a R-Fe-Co-B system permanent magnetic powder produced from an ingot of an alloy containing no Ti, V, Nb, Ta, Al, or Si was placed in a copper can in a vacuum, this was heated to a temperature of 720°C, and rolling was conducted a number of times so that the rolling ratio reached a value of 80%, and thus a conventional anisotropic magnet 4 was obtained.

[0050] The component compositions of the anisotropic magnets 120 - 164 of the present invention, the comparative anisotropic magnets 43 - 56, and the conventional anisotropic magnet 4 obtained in the above manner are shown in Tables 30 - 35. In addition, the average crystallized grain diameter, the amount (volume %) of crystallized grains present having a form in which the value of the ratio of the largest grain diameter / smallest grain diameter is less than 2, the magnetic characteristics, and the coercive force temperature coefficient αiHc of these anisotropic magnets were determined by a method identical to that given above, and the resulting values are shown in Tables 36 - 40.

[0051] From the results of Tables 36 - 40, it is dear that the R-Fe-Co-B system anisotropic magnets 120 - 164 of the present invention containing one or a plurality of Ti, V, Nb, Ta, Al, and Si, have essentially identical magnetic characteristics when compared with the conventional anisotropic magnet 4, which does not contain these elements; however, the coercive force temperature coefficient is markedly smaller. Furthermore, when the contained amount of Ti, V, Nb, Ta, Al, and Si lies outside the ranges of the present invention, as in the case of the comparative anisotropic magnets 43 - 56, the magnetic anisotropy worsens, and it is clear that the crystallized grain diameter and crystallized grain form also have a large influence on the magnetic characteristics.

TABLE 30

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)													
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT							Fe
								Ti	V	Nb	Ta	Al	Si		
ANISOTROPIC ELEMENTS OF THE PRESENT INVENTION	120	12.0	–	0.4	–	11.6	6.1	0.01	–	–	–	–	–	0.01	*
	121	12.1	–	0.4	–	11.6	6.0	0.1	–	–	–	–	–	0.1	*
	122	12.1	–	0.4	–	11.6	6.0	0.6	–	–	–	–	–	0.6	*
	123	12.1	–	0.4	–	11.4	6.1	5.0	–	–	–	–	–	5.0	*
	124	12.1	–	0.4	–	11.5	6.1	–	0.05	–	–	–	–	0.05	*
	125	12.1	–	0.4	–	11.5	6.1	–	0.1	–	–	–	–	0.1	*
	126	12.0	–	0.4	–	11.5	6.1	–	1.0	–	–	–	–	1.0	*
	127	12.0	–	0.4	–	11.6	6.1	–	4.0	–	–	–	–	4.0	*
	128	12.0	–	–	0.5	11.6	6.0	–	–	0.01	–	–	–	0.01	*
	129	12.0	–	–	0.6	11.6	6.1	–	–	0.3	–	–	–	0.3	*
	130	12.0	–	–	0.5	11.5	6.0	–	–	1.0	–	–	–	1.0	*
	131	12.0	–	–	0.6	11.6	6.1	–	–	5.0	–	–	–	5.0	*

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TABLE 31

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)													
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT							Fe
								Ti	V	Nb	Ta	Al	Si		
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	132	12.2	0.2	—	—	11.6	6.0	—	—	—	0.02	—	—	0.02	*
	133	12.2	0.2	—	—	11.6	5.9	—	—	—	0.3	—	—	0.3	*
	134	12.2	0.3	—	—	11.6	6.1	—	—	—	2.3	—	—	2.3	*
	135	12.1	0.3	—	—	11.6	6.0	—	—	—	3.8	—	—	3.8	*
	136	12.6	—	—	—	11.5	6.0	—	—	—	—	0.01	—	0.01	*
	137	12.5	—	—	—	11.5	6.1	—	—	—	—	0.5	—	0.5	*
	138	12.4	—	—	—	11.6	5.9	—	—	—	—	1.0	—	1.0	*
	139	12.4	—	—	—	11.7	6.1	—	—	—	—	4.9	—	4.9	*
	140	12.0	—	—	0.5	11.6	6.0	—	—	—	—	—	0.04	0.04	*
	141	12.1	—	—	0.4	11.6	6.0	—	—	—	—	—	0.5	0.5	*
	142	12.1	—	—	0.4	11.6	6.0	—	—	—	—	—	1.0	1.0	*
	143	12.0	—	—	0.5	11.7	6.1	—	—	—	—	—	3.6	3.6	*

* balance

TABLE 32

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)													
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT							Fe
								Ti	V	Nb	Ta	Al	Si		
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	144	12.3	–	–	–	17.4	6.5	–	–	0.3	–	–	1.5	1.8	*
	145	12.2	–	–	–	5.6	6.5	–	–	–	0.3	0.1	1.0	1.4	*
	146	12.3	–	–	–	17.3	6.4	–	0.1	0.2	–	0.5	2.0	2.8	*
	147	12.3	–	–	–	11.6	6.4	0.2	–	0.3	–	2.0	1.5	4.0	*
	148	12.3	–	–	–	5.8	6.5	0.1	–	0.3	0.2	2.5	1.0	4.1	*
	149	12.2	–	–	–	17.4	6.5	–	–	–	0.3	–	0.5	0.8	*
	150	12.2	–	–	–	11.5	6.4	–	–	0.1	0.3	0.2	0.2	0.8	*
	151	12.0	–	–	0.4	11.5	6.5	–	–	0.2	0.1	0.5	0.5	1.3	*
	152	12.0	–	–	0.3	11.5	6.5	0.1	0.1	–	–	0.5	0.5	1.2	*
	153	12.1	–	–	0.4	11.6	6.5	–	0.1	0.3	–	0.3	0.5	1.2	*
	154	11.9	–	–	0.1	11.6	6.4	0.1	0.1	0.1	0.1	0.1	0.1	0.6	*

* balance

TABLE 33

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)													
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT							Fe
								Ti	V	Nb	Ta	Al	Si		
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	155	13.0	–	0.7	–	11.5	6.4	–	–	0.3	–	0.5	0.5	1.3	*
	156	10.0	–	–	–	5.8	7.0	–	–	0.2	0.1	–	1.0	1.3	*
	157	14.0	–	–	–	11.6	7.2	–	–	0.2	0.1	–	1.0	1.3	*
	158	20.0	–	–	–	17.4	7.2	–	–	0.2	0.1	–	1.0	1.3	*
	159	15.5	–	–	0.5	11.5	3.0	–	–	–	0.2	0.5	0.5	1.2	*
	160	13.5	–	–	0.5	11.5	10.0	–	–	–	0.3	0.7	–	1.0	*
	161	14.0	–	–	0.5	11.5	20.0	–	–	0.1	0.1	0.2	0.5	0.9	*
	162	12.0	0.5	–	–	5.3	7.0	–	–	0.3	–	–	1.0	1.3	*

* balance

TABLE 34

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)													
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT							Fe
								Ti	V	Nb	Ta	Al	Si		
ANISOTROPIC MAGNETS OF PRESENT INVENTION	163	12.0	0.5	–	–	30.0	7.0	–	–	0.3	–	–	1.0	1.3	*
	164	12.0	0.5	–	–	46.2	7.0	–	–	0.3	–	–	1.0	1.3	*
COMPARATIVE ANISOTROPIC MAGNETS	43	12.2	–	–	0.3	11.5	6.0	0.1	–	0.1	–	0.1	0.1	0.4	*
	44	13.0	–	1.5	–	11.6	7.5	–	0.2	–	0.2	–	–	0.4	*
	45	12.2	–	–	0.3	11.5	6.1	7.0※	–	–	–	–	–	7.0※	*
	46	12.2	–	–	0.3	11.6	5.9	–	7.0※	–	–	–	–	7.0※	*
	47	12.1	–	–	0.3	11.6	6.0	–	–	7.1※	–	–	–	7.1※	*
	48	12.2	–	–	0.2	11.6	6.0	–	–	–	7.1※	–	–	7.1※	*
	49	12.1	–	–	0.3	11.6	6.1	–	–	–	–	6.5※	–	6.5※	*

(※ INDICATES VALUES WHICH DO NOT FULFILL THE CONDITIONS OF THE PRESENT INVENTION)

*balance

TABLE 35

CLASSIFICATION		COMPONENT ELEMENTS (ATOMIC %)													
		Nd	Tb	Dy	Pr	Co	B	TOTAL AMOUNT							Fe
								Ti	V	Nb	Ta	Al	Si		
COMPARATIVE ANISOTROPIC MAGNETS	50	12.2	–	–	0.3	11.5	6.0	–	–	–	–	–	7.1※	7.1※	*
	51	12.3	–	–	0.3	11.6	6.1	2.5	2.5	2.5	2.5	–	–	10.0※	*
	52	9.0※	–	–	–	11.5	7.0	–	0.5	–	0.5	–	0.5	1.5	*
	53	25.0※	–	–	–	11.4	7.1	–	–	–	–	1.0	1.0	2.0	*
	54	12.5	–	–	–	52.0※	7.0	2.5	–	–	–	–	1.0	3.5	*
	55	15.5	–	–	–	11.3	2.0※	–	0.3	–	–	1.0	–	1.3	*
	56	15.1	–	–	–	11.4	21.0※	–	0.3	–	–	1.0	–	1.3	*
CONVENTIONAL ANISOTROPIC MAGNET 4		14.0	–	–	–	11.6	7.2	–	–	–	–	–	–	–	*

(※INDICATES VALUES WHICH DO NOT FULFILL THE CONDITIONS OF THE PRESENT INVENTION)

* balance

TABLE 36

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μ m)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α I H c (% / °C)	MAGNETIC CHARACTERISTICS		
					B r (KG) 10^{-1}T	I H c (Koe) kA/m	B H max (MGOe) kJ/m^3
ANISOTROPIC MAGNETS IN ACCORDANCE WITH THE PRESENT INVENTION	120	0.5	90	-0.52	10.7	928 (11.6)	203.2 (25.4)
	121	0.2	90	-0.51	11.3	920 (11.5)	228.8 (28.6)
	122	0.2	100	-0.49	11.9	880 (11.0)	252 (31.5)
	123	0.1	90	-0.49	10.7	824 (10.3)	205.6 (25.7)
	124	0.5	90	-0.51	10.6	984 (12.3)	200 (25.0)
	125	0.2	80	-0.49	11.4	944 (11.8)	228.4 (28.3)
	126	0.3	90	-0.48	11.2	840 (10.5)	220 (27.5)
	127	0.2	90	-0.48	10.9	808 (10.1)	202.4 (25.3)
	128	0.4	90	-0.51	11.8	1000 (12.5)	240 (30.0)
	129	0.3	90	-0.49	12.9	928 (11.6)	282.4 (35.3)
	130	0.2	100	-0.49	12.5	880 (11.0)	268.8 (33.6)
	131	0.3	80	-0.48	11.0	824 (10.3)	208.8 (26.1)
	132	0.2	90	-0.50	11.1	928 (11.6)	212 (26.5)
	133	0.4	90	-0.50	12.8	936 (11.7)	290.4 (36.3)
	134	0.1	80	-0.51	12.0	816 (10.2)	257.6 (32.2)

TABLE 37

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μ m)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α I H c (%/°C)	MAGNETIC CHARACTERISTICS		
					B r (KG) 10^{-1}T	I H c (Koe) kA/m	B H max (MGOe) kJ/m^3
ANISOTROPIC MAGNETS OF THE PRESENT INVENTION	135	0.05	80	-0.48	11.3	808 (10.1)	224.8 (28.1)
	136	0.3	80	-0.52	11.4	1008 (12.6)	226.4 (28.3)
	137	0.5	80	-0.52	12.8	1144 (14.3)	289.6 (36.2)
	138	0.5	60	-0.52	12.0	1120 (14.0)	257.6 (32.2)
	139	0.5	70	-0.51	11.8	976 (12.2)	247.2 (30.9)
	140	0.3	90	-0.50	11.7	960 (12.0)	244 (30.5)
	141	0.2	100	-0.49	13.0	1208 (15.1)	320.8 (40.1)
	142	0.1	80	-0.48	12.6	1176 (14.7)	284 (35.5)
	143	0.1	90	-0.50	12.4	1064 (13.3)	274.4 (34.3)
	144	0.2	90	-0.49	13.1	1040 (13.0)	316 (39.5)
	145	0.2	80	-0.49	13.2	1072 (13.4)	328 (41.0)
	146	0.3	90	-0.51	11.8	1032 (12.9)	260 (32.5)
	147	0.5	100	-0.50	11.7	928 (11.6)	252.8 (31.6)
	148	0.3	80	-0.50	11.6	1024 (12.8)	248 (31.0)
	149	0.2	90	-0.48	13.3	1088 (13.6)	332 (41.5)

TABLE 38

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μm)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α I H c (%/°C)	MAGNETIC CHARACTERISTICS		
					Br (KG) 10^{-1}T	I H c (Koe) kA/m	B H max (MGOe) kJ/m^3
ANISOTROPIC MAGENTS OF THE PRESENT INVENTION	150	0.3	80	-0.48	13.2	1080 (13.5)	320.8 (40.1)
	151	0.5	70	-0.50	12.8	1120 (14.0)	291.2 (36.4)
	152	0.2	90	-0.51	12.4	1096 (13.7)	268 (33.5)
	153	0.2	90	-0.48	13.0	1088 (13.6)	309.6 (38.7)
	154	0.3	90	-0.51	12.6	1016 (12.7)	280 (35.0)
	155	1.0	50	-0.52	12.4	704 (8.8)	260 (32.5)
	156	0.2	100	-0.53	11.2	776 (9.7)	209.6 (26.2)
	157	0.3	90	-0.51	12.6	1136 (14.2)	280.8 (35.1)
	158	2.0	70	-0.53	10.9	784 (9.8)	206.4 (25.8)
	159	0.3	90	-0.52	10.7	712 (8.9)	193.6 (24.2)
	160	1.0	80	-0.50	11.8	832 (10.4)	241.6 (30.2)
	161	2.0	80	-0.53	11.0	736 (9.2)	204 (25.5)

TABLE 39

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μm)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α iHc (%/°C)	MAGNETIC CHARACTERISTICS		
					Br (KG) 10^{-1}T	iHc (Koe) kA/m	BHmax (MGOe) kJ/m^3
ANISOTROPIC MAGNETS OF PRESENT INVENTION	162	0.4	80	-0.51	12.5	1048 (13.1)	268 (33.5)
	163	0.2	60	-0.47	11.6	848 (10.6)	228 (28.5)
	164	0.2	80	-0.45	11.0	672 (8.4)	196 (24.5)
COMPARATIVE ANISOTROPIC MAGNETS	43	0.01※	90	-0.50	9.2	248 (3.1)	43.2 (5.4)
	44	22※	90	—	7.3	96 (1.2)	(< 5) < 40
	45	0.5	80	-0.52	9.7	272 (3.4)	48 (6.0)
	46	0.3	80	-0.52	8.6	248 (3.1)	40.8 (5.1)
	47	0.3	90	-0.52	9.0	240 (3.0)	40 (5.0)
	48	0.5	90	—	8.9	216 (2.7)	40 (5.0)
	49	0.3	80	-0.52	9.1	264 (3.3)	46.4 (5.8)

(※ INDICATES VALUES WHICH DID NOT FULFILL THE CONDITIONS OF THE PRESENT INVENTION)

TABLE 40

CLASSIFICATION		AVERAGE CRYSTAL GRAIN DIAMETER (μ m)	AMOUNT OF CRYSTAL GRAINS PRESENT FOR WHICH THE LARGEST GRAIN DIAMETER/ SMALLEST GRAIN DIAMETER IS LESS THAN 2 (VOLUME %)	COERCIVE FORCE TEMPERATURE COEFFICIENT α_{IHc} (% / °C)	MAGNETIC CHARACTERISTICS		
					Br (KG) 10^{-1}T	I Hc (Koe) kA/m	B H max (MGOe) kJ/m^3
COMPARATIVE ANISOTROPIC MAGNETS	50	1.0	80	-0.53	8.0	272 (3.4)	(< 5) < 40
	51	0.2	80	-0.53	9.7	240 (3.0)	41.6 (5.2)
	52	1.0	80	–	7.3	56 (0.7)	(< 5) < 40
	53	3.0	80	-0.55	7.0	352 (4.4)	(< 5) < 40
	54	2.0	70	–	8.8	184 (2.3)	(< 5) < 40
	55	1.0	60	–	7.3	80 (1.0)	(< 5) < 40
	56	1.0	80	-0.54	6.5	280 (3.5)	(< 5) < 40
CONVENTIONAL ANISOTROPIC MAGNET 4		0.8	40※	-0.71	12.1	1000 (12.5)	260 (32.5)

(※ INDICATES VALUES WHICH DO NOT FULFILL THE CONDITIONS OF THE PRESENT INVENTION)

POSSIBILITIES FOR USE IN INDUSTRY

[0052] In accordance with the present invention, by means of using a hydrogen treated powder of a R-Fe-Co-B system containing one or a plurality of Ga, Zr, and Hf, or containing one or a plurality of Ti, V, Nb, Ta, Al, and Si, it is possible to obtain an anisotropic magnet for which the magnetic anisotropy is large, and moreover, the coercive force temperature coefficient is small, so that there is no need to conduct a magnetic anisotropizing process such as a thermoplastic process or the like, as in the conventional method, and production costs can be thereby reduced considerably. Accordingly, the invention contributes greatly to the ability to produce electronic machines such as motors and the like and to improvements in stability.

Claims

1. A method for preparing a Rare Earth-Fe-Co-B system anisotropic magnet which is a hot press moulded body or a hot isostatic press moulded body comprising:

a rare earth element including Y,
B,
0.001-5 atomic% in total of one or more of Ga, Zr and Hf, and
0.1-50 atomic% Co;
with the remainder being Fe and unavoidable impurities; said body having an aggregate structure of crystallised grains comprising materially only a phase of an $R_2(TM)_{14}B$ type intermetallic compound with a tetragonal structure, wherein R is at least one rare earth element including Y, and TM is Fe and Co, the crystallised grains having dimensions of 0.05-20 μm; and the ratio of the largest grain diameter b to the smallest grain diameter a is less than two for individual crystallised grains constituting more than 50 volume % of the total crystallised grains of the aggregate structure,

the method comprising the steps of:

(i) heating a R-Fe-Co-B mother alloy including one or more of Ga, Zr and Hf in an atmosphere of hydrogen gas, optionally including an inert gas, at 500-1000°C,
(ii) removing hydrogen from the atmosphere at a temperature of 500-1000°C so as to produce a vacuum atmosphere having a hydrogen gas pressure of less than 0.13 kPa (less than 1 Torr) or an inert gas atmosphere in which the partial pressure of hydrogen gas is less than 0.13 kPa (less than 1 Torr),
(iii) cooling the alloy to obtain a R-Fe-Co-B system permanent magnetic powder having a recrystallised aggregate structure comprising materially only $R_2(Fe,Co)_{14}B$ type intermetallic compound phase,
(iv) pressing the magnetic powder into a green compact in a magnetic field, and
(v) subjecting the green compact to hot press moulding or hot isostatic press moulding at a temperature of 600-900°C.

2. A method according to Claim 1, wherein the magnet comprises a total of 0.01-2.0 atomic% of one or a plurality of Al, V and Si.

3. A method for preparing a Rare Earth-Fe-Co-B system anisotropic magnet which is a hot press moulded body or a hot isostatic press moulded body comprising:

a rare earth element including Y,
B,
0.001-5 atomic% in total of one or more of Ti, V, Nb, Ta, Al and Si, and
0.1-50 atomic% Co;
with the remainder being Fe and unavoidable impurities; said body having an aggregate structure of crystallised grains comprising materially only a phase of an $R_2(TM)_{14}B$ type intermetallic compound with a tetragonal structure, wherein R is at least one rare earth element including Y, and TM is Fe and Co, the crystallised grains having dimensions of 0.05-20 μm ; and the ratio of the largest grain diameter b to the smallest grain diameter a is less than two for individual crystallised grains constituting more than 50 volume % of the total crystallised grains of the aggregate structure,

the method comprising the steps of:

(i) heating a R-Fe-Co-B mother alloy including one or more of Ti, V, Nb, Ta, Al and Si in an atmosphere of hydrogen gas, optionally including an inert gas, at 500-1000°C,

(ii) removing hydrogen from the atmosphere at a temperature of 500-1000°C so as to produce a vacuum atmosphere having a hydrogen gas pressure of less than 0.13 kPa (less than 1 Torr) or an inert gas atmosphere in which the partial pressure of hydrogen gas is less than 0.13 kPa (less than 1 Torr),

(iii) cooling the alloy to obtain a R-Fe-Co-B system permanent magnetic powder having a recrystallised aggregate structure comprising materially only $R_2(Fe,Co)_{14}B$ type intermetallic compound phase,

(iv) pressing the magnetic powder into a green compact in a magnetic field, and

(v) subjecting the green compact to hot press moulding or hot isostatic press moulding at a temperature of 600-900°C.

4. A method according to any preceding Claim, wherein the average diameter of the crystallised grains is 0.05-3 µm.

Patentansprüche

1. Verfahren zur Herstellung eines anisotropen Magneten vom Seltenerd-Fe-Co-B-System, der ein heißgepreßter Formkörper oder ein heißisostatisch gepreßter Formkörper ist und umfaßt:

ein Seltenerdelement einschließlich Y,
B,
0,001 - 5 Atom% insgesamt von einem oder mehreren Elementen aus Ga, Zr und Hf, und
0,1 - 50 Atom% Co;

wobei der Rest Fe und unvermeidbare Verunreinigungen sind;
wobei dieser Körper eine Aggregatstruktur von kristallisierten Körnern hat, die im wesentlichen nur eine Phase einer intermetallischen Verbindung vom $R_2(TM)_{14}B$-Typ mit einer tetragonalen Struktur umfaßt, worin R mindestens ein Seltenerdelement einschließlich Y ist und TM Fe und Co ist, und die kristallisierten Körner Dimensionen von 0,05 - 20 µm haben; und das Verhältnis des größten Korndurchmessers b zum kleinsten Korndurchmesser a kleiner ist als zwei für individuelle kristallisierte Körner, die mehr als 50 Vol.% der gesamten kristallisierten Körner der Aggregatstruktur darstellen;
wobei das Verfahren die Schritte umfaßt:

(i) Erwärmen einer R-Fe-Co-B-Mutterlegierung einschließlich eines oder mehrerer Elemente aus Ga, Zr und Hf in einer Wasserstoffgasatmosphäre, die gegebenenfalls ein Inertgas einschließt, bei 500 bis 1.000°C,

(ii) Entfernen von Wasserstoff aus der Atmosphäre bei einer Temperatur von 500 bis 1.000°C, um so eine Vakuumatmosphäre mit einem Wasserstoffgasdruck von weniger als 0,13 kPa (weniger als 1 Torr) oder eine Inertgasatmosphäre, in der der Partialdruck von Wasserstoffgas weniger als 0,13 kPa (weniger als 1 Torr) beträgt, zu erzeugen,

(iii) Abkühlen der Legierung, um ein permanentmagnetisches Pulver vom R-Fe-Co-B-System zu erhalten mit einer rekristallisierten Aggregatstruktur, die im wesentlichen nur eine Phase einer intermetallischen Verbindung vom $R_2(Fe,Co)_{14}B$-Typ umfaßt,

(iv) Pressen des magnetischen Pulvers in einem magnetischen Feld in einen Grünkörper, und

(v) Heißformpressen oder heißisostatisches Formpressen des Grünkörpers bei einer Temperatur von 600 bis 900°C.

2. Verfahren gemäß Anspruch 1, wobei der Magnet insgesamt 0,01 - 2,0 Atom% eines oder mehrerer Elemente aus Al, V und Si umfaßt.

3. Verfahren zur Herstellung eines anisotropen Magneten vom Seltenerd-Fe-Co-B-System, der ein heißgepreßter Formkörper oder ein heißisostatisch gepreßter Formkörper ist und umfaßt:

ein Seltenerdelement einschließlich Y,
B,

0,001 - 5 Atom% insgesamt von einem oder mehreren Elementen aus Ti, V, Nb, Ta, Al und Si, und
0,1 - 50 Atom% Co;

wobei der Rest Fe und unvermeidbare Verunreinigungen sind; wobei dieser Körper eine Aggregatstruktur von kristallisierten Körnern hat, die im wesentlichen nur eine Phase einer intermetallischen Verbindung vom $R_2(TM)_{14}B$-Typ mit einer tetragonalen Struktur umfaßt, worin R mindestens ein Seltenerdelement einschließlich Y ist und TM Fe und Co ist, und die kristallisierten Körner Dimensionen von 0,05 - 20 µm haben; und das Verhältnis des größten Korndurchmessers b zum kleinsten Korndurchmesser a kleiner ist als zwei für individuelle kristallisierte Körner, die mehr als 50 Vol.% der gesamten kristallisierten Körner der Aggregatstruktur ausmachen,
wobei das Verfahren die Schritte umfaßt:

(i) Erwärmen einer R-Fe-Co-B-Mutterlegierung einschließlich eines oder mehrerer Elemente aus Ti, V, Nb, Ta, Al und Si in einer Wasserstoffgasatmosphäre, die gegebenenfalls ein Inertgas einschließt, bei 500 bis 1.000°C,

(ii) Entfernen von Wasserstoff aus der Atmosphäre bei einer Temperatur von 500 bis 1.000°C, um so eine Vakuumatmosphäre mit einem Wasserstoffgasdruck von weniger als 0,13 kPa (weniger als 1 Torr) oder eine Inertgasatmosphäre, in der der Partialdruck von Wasserstoffgas weniger als 0,13 kPa (weniger als 1 Torr) beträgt, zu erzeugen,

(iii) Abkühlen der Legierung, um ein permanentmagnetisches Pulver vom R-Fe-Co-B-System zu erhalten mit einer rekristallisierten Aggregatstruktur, die im wesentlichen nur eine Phase einer intermetallischen Verbindung vom $R_2(Fe,Co)_{14}B$-Typ umfaßt,

(iv) Pressen des magnetischen Pulvers in einem magnetischen Feld in einen Grünkörper, und

(v) Heißformpressen oder heißisostatisches Formpressen des Grünkörpers bei einer Temperatur von 600 bis 900°C.

4. Verfahren gemäß einem der vorstehenden Ansprüche, wobei der durchschnittliche Durchmesser der kristallisierten Körner 0,05 bis 3 µm ist.

Revendications

1. Une méthode afin de préparer un aimant anisotrope du système Terre Rare-Fe-Co-B qui est un corps moulé sous compression à chaud ou un corps moulé sous compression isostatique à chaud comprenant :

un élément de terre rare incluant Y,
B,
0,001-5% d'atomes au total d'un ou de plusieurs de Ga, Zr et Hf, et
0,1-50% d'atomes de Co ;
avec le reste étant du Fe et des impuretés inévitables ; ledit corps ayant une structure d'agrégat de grains cristallisés comprenant, matériellement, seulement une phase d'un composé intermétallique du type $R_2(TM)_{14}B$ avec une structure tétragonale, où R est au moins un élément des terres rares incluant Y, et TM est Fe et Co, les grains cristallisés ayant une dimension de 0,05-20 µm ; et le rapport entre le diamètre b le plus grand des grains et le diamètre a le plus petit des grains est inférieur à deux pour des grains individuels cristallisés constituant plus de 50 % en volume des grains cristallisés totaux de la structure d'agrégat,

la méthode comprenant les étapes de :

(i) chauffer un alliage mère R-Fe-Co-B incluant un ou plusieurs de Ga, Zr et Hf dans une atmosphère d'hydrogène gazeux incluant optionnellement un gaz inerte, à 500-1000 °C,
(ii) enlever l'hydrogène de l'atmosphère à une température de 500-1000°C de manière à produire une atmosphère de vide ayant une pression d'hydrogène gazeux plus petite que 0,13 kPa (moins que 1 Torr) ou une atmosphère de gaz inerte dans laquelle la pression partielle d'hydrogène gazeux est plus petite que 0,13 kPa (moins que 1 Torr),
(iii) refroidir l'alliage afin d'obtenir une poudre magnétique permanente d'un système R-Fe-Co-B ayant une

structure d'agrégat recristallisée comprenant matériellement qu'une phase de composé intermétallique du type $R_2(Fe,Co)_{14}B$.
(iv) presser la poudre magnétique en un comprimé vert dans un champs magnétique, et
(v) soumettre le comprimé vert à un moulage sous presse chaude ou à un moulage sous presse isostatique chaude à une température de 600-900°C.

2. Une méthode selon la revendication 1, dans laquelle l'aimant comprend un total de 0,01-2,0% d'atomes d'un ou d'une multitude de Al, V et Si.

3. Une méthode afin de préparer un aimant anisotrope du système Terre Rare-Fe-Co-B qui est un corps moulé sous compression à chaud ou un corps moulé sous compression isostatique à chaud comprenant :

un élément de terre rare incluant Y,
B,
0,001-5% d'atomes au total d'un ou de plusieurs de Ti, V, Nb, Ta, Al et Si et
0,1-50% d'atomes de Co ;
avec le reste étant du Fe et des impuretés inévitables ; ledit corps ayant une structure d'agrégat de grains cristallisés comprenant, matériellement, seulement une phase d'un composé intermétallique du type $R_2(TM)_{14}B$ avec une structure tétragonale, où R est au moins un élément des terres rares incluant Y, et TM est Fe et Co, les grains cristallisés ayant une dimension de 0.05-20 µm ; et le rapport entre le diamètre b le plus grand des grains et le diamètre a le plus petit des grains est inférieur à deux pour des grains individuels cristallisés constituant plus de 50 % en volume des grains cristallisés totaux de la structure d'agrégat,

la méthode comprenant les étapes de :

(i) chauffer un alliage mère R-Fe-Co-B incluant un ou plusieurs de Ti, V, Nb, Ta, Al et Si dans une atmosphère d'hydrogène gazeux incluant optionnellement un gaz inerte, à 500-1000 °C,
(ii) enlever l'hydrogène de l'atmosphère à une température de 500-1000°C de manière à produire une atmosphère de vide ayant une pression d'hydrogène gazeux plus petite que 0.13 kPa (moins que 1 Torr) ou une atmosphère de gaz inerte dans laquelle la pression partielle d'hydrogène gazeux est plus petite que 0.13 kPa (moins que 1 Torr),
(iii) refroidir l'alliage afin d'obtenir une poudre magnétique permanente d'un système R-Fe-Co-B ayant une structure agrégée recristallisée comprenant matériellement qu'une phase de composé intermétallique du type $R_2(Fe,Co)_{14}B$.
(iv) presser la poudre magnétique en un comprimé vert dans un champs magnétique, et
(v) soumettre le comprimé vert à un moulage sous presse chaude ou à un moulage sous presse isostatique chaude à une température de 600-900°C.

4. Une méthode selon n'importe laquelle des revendications précédentes, dans laquelle le diamètre moyen des grains cristallisés est 0.05-3 µm.