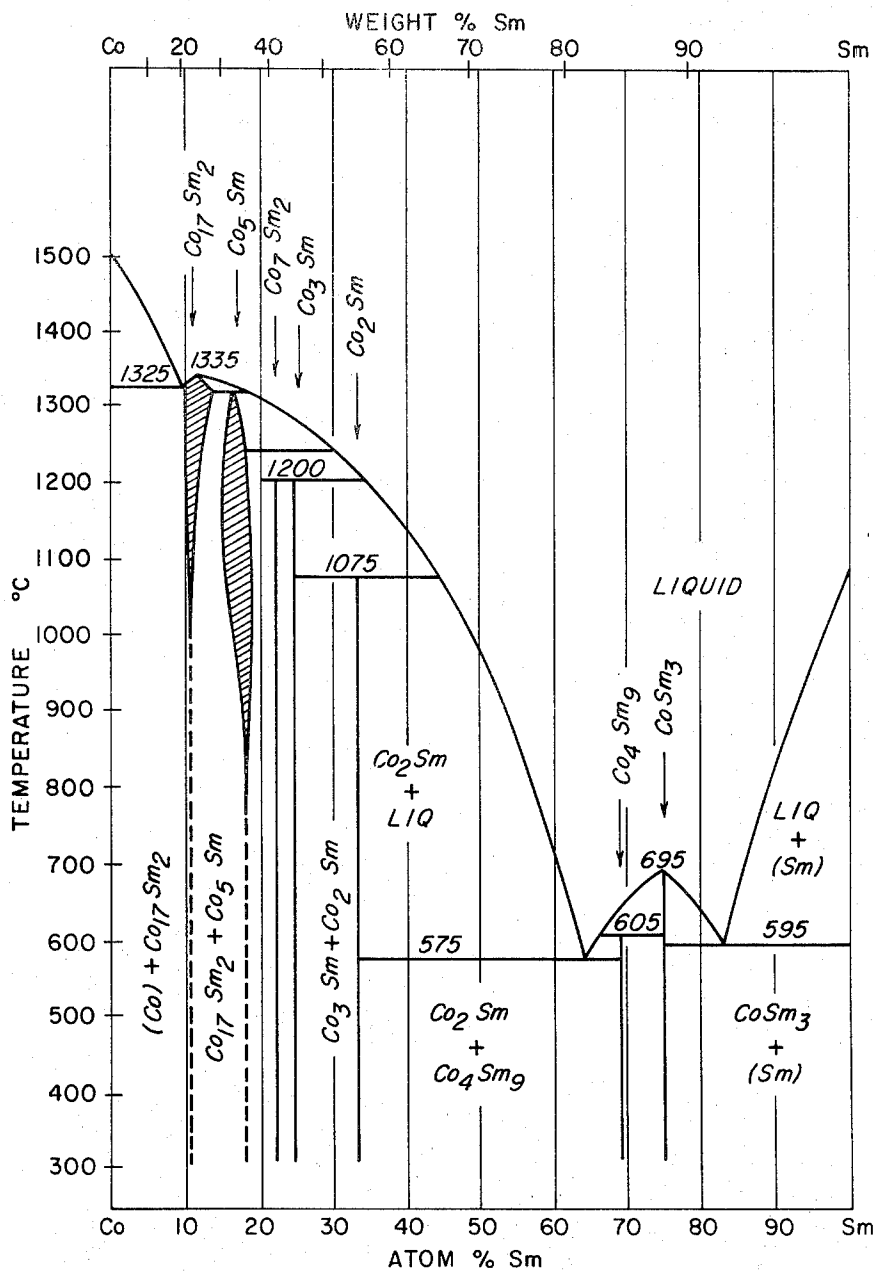


Aug. 15, 1972

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3,684 591

SINTERED COBALT-RARE EARTH INTERMETALLIC PRODUCT
INCLUDING SAMARIUM AND CERIU AND PERMANENT
MAGNETS PRODUCED THEREFROM
Filed Aug. 24, 1970



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3,684,591

SINTERED COBALT-RARE EARTH INTERMETALLIC PRODUCT INCLUDING SAMARIUM AND CERIUM AND PERMANENT MAGNETS PRODUCED THEREFROM

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Filed Aug. 24, 1970, Ser. No. 66,172

Int. Cl. H01f 1/08

U.S. Cl. 148—31.57

6 Claims

ABSTRACT OF THE DISCLOSURE

Novel sintered cobalt-rare earth intermetallic products are used to form permanent magnets having unique properties. The sintered product is comprised of intermetallic compounds of cobalt and rare earth metals composed of samarium and cerium. Cobalt is present in an amount of about 60 to 66 percent by weight of the product and the rare earth metals are present in an amount of about 34 to 40 percent by weight of the product with the cerium component ranging in amount from about 10 to 90 percent by weight of the rare earth content. Permanent magnets are formed from the sintered product in bulk form or in particulate form.

The present invention relates generally to the art of permanent magnets and is more particularly concerned with novel sintered cobalt-samarium-cerium intermetallic products having unique characteristics and with permanent magnets formed therefrom.

Permanent magnets, i.e. "hard" magnetic materials such as the cobalt-rare earth intermetallic compounds, are of technological importance because they can maintain a high, constant magnetic flux in the absence of an exciting magnetic field or electrical current to bring about such a field.

Cobalt-rare earth intermetallic compounds exist in a variety of phases. The permanent magnet properties of cobalt-rare earth intermetallic magnetic materials generally can be enhanced by reducing the bulk bodies to powders, but in such finely-divided form these materials are unstable in air and their magnetic properties deteriorate after a short period of time.

One object of the present invention is to provide novel cobalt-rare earth intermetallic magnets which are stable. The cobalt-rare earth intermetallic materials of the present invention are comprised of cobalt, samarium and cerium of specific composition.

Those skilled in the art will gain a further and better understanding of the present invention from the detailed description set forth below, considered in conjunction with the figure accompanying and forming a part of the specification, in which:

The accompanying figure is the cobalt-samarium phase diagram. It is assumed herein that the phase diagram at 300° C., which is the lowest temperature shown in the figure, is substantially the same at room temperatures.

Briefly stated, the sintered product of the present invention is comprised of intermetallic compounds of cobalt and rare earth metals composed of samarium and cerium. Cobalt is present in an amount of about 60 to 66

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percent by weight of the product and the rare earth metals are present in an amount of about 34 to 40 percent by weight of the product with the cerium component ranging in amount from about 10 to 90 percent by weight of the rare earth content. Permanent magnets are formed from the sintered product in bulk form or in particulate form.

The sintered product of the present invention may be produced in a variety of different ways but I prefer to use substantially the process disclosed and claimed in copending U.S. patent application Ser. No. 33,347, entitled "Liquid Sintered Cobalt-Rare Earth Intermetallic Product," filed on Apr. 30, 1970 in the name of Mark G. Benz, and assigned to the assignee hereof, and which by reference is made part of the disclosure of the present application. Briefly stated, the process of U.S. patent application Ser. No. 33,347 comprises the steps of forming a particulate mixture of a base cobalt-rare earth alloy and additive cobalt-rare earth alloy. The base alloy is one which at sintering temperature exists as a solid Co_3R intermetallic single phase where R is a rare earth metal. The additive cobalt-rare earth alloy is richer in rare earth metal than the base alloy, and at sintering temperature it is at least partly in liquid form and thus increases the sintering rate. The mixture is compacted to produce a green body which is sintered to the desired density and phase composition. The final sintered product contains a major amount of the Co_3R intermetallic phase and up to about 35 percent by weight of the product of a second solid CoR intermetallic phase which is richer in rare earth content than the Co_3R phase.

The sintered product of my invention is also suitably produced by using substantially the process disclosed and claimed in copending U.S. patent application Ser. No. 33,348 entitled "Sintered Cobalt-Rare Earth Intermetallic Product and Process Using Solid Sintering Additive," filed on Apr. 30, 1970 in the name of Mark G. Benz and assigned to the assignee hereof, and which by reference is made part of a disclosure of the present application.

The procedure for forming sintered products disclosed in U.S. patent application Ser. No. 33,348 is substantially the same as that disclosed in U.S. patent application Ser. No. 33,347 except that an additive CoR alloy which is solid at sintering temperature and which is richer in rare earth metal than the base alloy is used.

As applied to the preparation of the new products of the present invention, the process is carried out with a base alloy which is solid at sintering temperature and which at sintering temperature is comprised substantially or completely of Co_3R intermetallic phase where R is samarium, cerium, or preferably, a mixture of samarium and cerium. Generally, the present base alloy is comprised of about 65 to 70 percent by weight cobalt and about 30 to 35 percent by weight rare earth metal or metals. Although the base alloy may vary in composition, it should have a composition which together with the sintering additive, produces the claimed composition of the present sintered product.

The present sintering additive is a cobalt-rare earth metal alloy which is richer in rare earth metal content than the base alloy. Preferably, it is also one that exists at least partly in a liquid form at sintering temperature, but it can be a solid. Representative of the present sintering additives are alloys of cobalt-samarium, cobalt-cerium

um, and cobalt-samarium-cerium. In certain instances, it may be desirable for the product to contain additional rare earth metal components and this may be done by using a sintering additive which contains the desired rare earth metal component, such as, for example, cobalt-samarium-praseodymium-cerium and cobalt-lanthanum.

The sintering additive alloy may vary in composition and can be determined from the phase diagram for the particular cobalt-rare earth system or it can be determined empirically. When liquid phase sintering is desired, the accompanying figure shows that for the cobalt-samarium system, for example, there are phases which are partly or completely liquid at the temperature ranging from about 950 to 1200° C. Any alloy within the range shown in the figure which forms at least a partly liquid phase at the particular sintering temperature would be a satisfactory sintering additive. For example, as illustrated in the figure, the Co-Sm additive alloy can vary upward in samarium content from about 46 percent by weight of the additive.

When a sintering additive which is solid at sintering temperature is desired, it also may vary in composition and can be determined from the phase diagram for the particular cobalt-rare earth system or which can be determined empirically. For example, the accompanying figure shows that for the cobalt-samarium system, there is a solid phase containing samarium in an amount greater than about 36 percent by weight at a temperature ranging from 950 to 1200° C. Specifically, from a temperature of 950 to 1075° C., the solid additive alloy for the cobalt-samarium system ranges in samarium content from about 36 to about 55 percent by weight of the additive, and at temperatures ranging from 950 to 1200° C., the solid additive alloy may range in samarium content from about 36 percent to about 45 percent by weight of the additive. Any additive alloy within these ranges would be a satisfactory sintering additive alloy.

If desired, the sintering additive can be empirically selected by a number of methods, such as by means of a composition scan at the sintering temperature, i.e. heating samples of various additive alloy compositions to the desired sintering temperature to determine which is solid and which is at least partly liquid at sintering temperatures.

Although suitable sintering additive alloys fall within a general composition range, the preferred ones are comparatively low in rare earth metal content so that undesirable characteristics of the pure rare earth metal in the additive alloy are minimized. Specifically, for example, pure samarium is both pyrophoric and very ductile and consequently difficult to crush and to blend with the base alloy since it has a tendency to separate out and fall to the bottom of the container. However, a sintering additive Co-Sm alloy of samarium content preferably less than 70 percent by weight is substantially non-reactive at room temperature in air, it can be crushed by conventional techniques, and being slightly magnetic, it clings to the base alloy resulting in a substantially thorough stable mixture. The higher the cobalt content of the additive alloy, the stronger are its magnetic properties and the more stable is the particulate mixture it forms with the base alloy.

In preparing the present sintered product, the base and sintering additive cobalt-rare earth alloys can be formed by a number of methods. For example, each can be prepared by arc or induction melting the cobalt and rare earth metal together in the proper amounts under a substantially inert atmosphere such as argon and allowing the melt to solidify. Preferably, the melt is cast into an ingot.

The solid base and additive alloys can be converted to particulate form in a conventional manner. Such conversion can be carried out in air at room temperature since the alloys are substantially non-reactive. For ex-

ample, each alloy can be crushed by mortar and pestle in air and then pulverized to a finer form by fluid energy milling in a substantially inert atmosphere.

The particle size of the base and additive cobalt-rare earth alloys used in forming the present mixture may vary. Each can be in as finely divided a form as desired. For most applications, average particle size will range from about 1 micron or less to about 10 microns. Larger sized particles can be used, but as the particle size is increased, the maximum coercive force obtainable is lower because the coercive force generally varies inversely with particle size. In addition, the smaller the particle size, the lower is the sintering temperature which may be used.

In forming the present mixture, the base and sintering additive alloys are each used in an amount so that the resulting mixture has a cobalt and rare earth metal content substantially corresponding to that of the final desired sintered product. In addition, however, in forming the mixture, the sintering additive should be used in an amount sufficient to promote sintering. This amount depends largely on the specific composition of the additive and can be determined empirically, but generally, the sintering additive alloy should be used in an amount of at least 0.5 percent by weight of the base-additive alloy mixture. Generally, for liquid phase sintering, the larger the rare earth metal component of the sintering additive alloy, the more liquid it is at sintering temperature. Specifically, for liquid phase sintering, a sintering additive composed of 40 percent Co and 60 percent Sm may generally be used in an amount ranging from about 4 to 25 percent by weight of the base-additive alloy mixture wherein the base alloy is comprised of about 65 to 70 percent by weight cobalt and 30 to 35 percent by weight samarium and cerium.

In carrying out the process of this invention, the base alloy is admixed with the additive alloy in any suitable manner to produce a substantially thorough particulate mixture. The particulate mixture can then be compressed into a green body of the desired size and density by any of a number of techniques such as hydrostatic pressing or methods employing steel dies. Preferably, the mixture is compressed in the presence of an aligning magnetizing field to magnetically align the particles along there if desired, the mixture may be compressed after magnetically aligning the particles. The greater the magnetic alignment of the particles, the better are the resulting magnetic properties. Preferably, also, compression is carried out to produce a green body with as high a density as possible, since the higher its density, the greater the sintering rate. Green bodies having a density of about 40 percent or higher of theoretical are preferred.

The green body is sintered to produce a sintered body of desired density. Preferably, the green body is sintered to produce a sintered body wherein the pores are substantially non-interconnecting. Such non-interconnectivity stabilizes the permanent magnet properties of the product because the interior of the sintered product or magnet is protected against exposure to the ambient atmosphere.

The sintering temperature used in the present process may vary. The minimum sintering temperature must be sufficiently high for sintering to occur in a particular cobalt-rare earth system, i.e. it must be high enough to coalesce the component particles. In the present process, a sintering temperature of about 1000° C. to 1150° C. is suitable with a sintering temperature of 1050° C. to 1100° C. being particularly satisfactory.

Preferably, sintering is carried out so that the pores in the sintered product are substantially non-interconnecting. A sintered body having a density or packing of at least about 87 percent of theoretical is generally one wherein the pores are substantially non-interconnecting. Such non-interconnectivity is determinable by standard metallographic techniques, as for example, by means of transmission electron micrographs of a cross-section of the sintered product. The maximum sintering temperature is

preferably one at which significant growth of the component particles or grains does not occur, since too large an increase in grain size deteriorates magnetic properties such as coercive force. The green body is sintered in a substantially inert atmosphere such as argon, and upon completion of the sintering, it is preferably cooled to room temperatures in a substantially inert atmosphere.

The density of the sintered product may vary. The particular density depends largely on the particular permanent magnet properties desired. Preferably, to obtain a product with substantially stable permanent magnet properties, the density of the sintered product should be one wherein the pores are substantially non-interconnecting and this occurs usually at a density or packing of about 87 percent. Generally, for a number of applications, the density may range from about 80 percent to 100 percent. For example, for low temperature applications, a sintered body having a density ranging down to about 80 percent may be satisfactory. The preferred density of the sintered product is one which is the highest obtainable without producing a growth in grain size which would deteriorate magnetic properties significantly, since the higher the density the better are the magnetic properties. For sintered products of the present invention, a density of at least about 87 percent of theoretical, i.e. of full density, and as high as about 96 percent of theoretical is preferred to produce permanent magnets with suitable magnetic properties which are substantially stable.

Sintering of the green body produces a sintered product which weights about the same as the green body indicating no loss, or no significant loss, of cobalt and rare earth components. Standard chemical analysis of a sintered product shows that the rare earth and cobalt content is substantially unaffected by the sintering process.

Magnetization of the present sintered products of cobalt, samarium and cerium produces novel permanent magnets. Specifically, the magnetic properties of the present permanent magnets generally are significantly better than those provided by prior art magnets such as cobalt-platinum. One particular advantage of the present invention is that cerium is a much more abundant element than samarium or platinum, thereby making the present permanent magnets available for a wider variety of applications than has been possible heretofore.

The composition of the cobalt-rare earth alloy or alloys used to prepare the sintered product can be modified to substitute neodymium, or yttrium, or mixtures thereof, for a portion of the cerium component, as long as the minimum claimed amount of cerium is present in the final product composition, i.e. 10 percent by weight of the rare earth content, to produce additional novel sintered products and useful permanent magnets in the same manner as set forth herein. Specifically, where one of these additional rare earth metals is included, the present sintered product of modified composition would be cobalt-samarium-cerium-neodymium and cobalt-samarium-cerium-yttrium. In the resulting permanent magnets, the neodymium and yttrium components should increase the saturation induction B_s .

The permanent magnets of the present invention are substantially stable in air and have a wide variety of uses. For example, they are useful in telephones, electric clocks, radios, television, and phonographs. They are also useful in portable appliances, such as electric toothbrushes and to operate automobile accessories. In industrial equipment, the present permanent magnets can be used in such diverse applications as meters and instruments, magnetic separators, computers and microwave devices.

If desired, the sintered bulk product of the present invention can be crushed to a desired particle size preferably a powder, which is particularly suitable for alignment and matrix bonding to give a stable permanent magnet. The matrix material may vary widely and may be plastic, rubber or metal such as, for example, lead, tin, zinc, copper or aluminum. The powder-containing matrix

can be cast, pressed or extruded to form the desired permanent magnet.

All parts and percentages used herein are by weight unless otherwise noted.

The invention is further illustrated by the following examples in which, unless otherwise noted, the conditions and procedure were as follows:

The base alloy and sintering addition compositions as well as the compositions of the green bodies were determined on a nominal weight bases.

Alignment is the ratio of the magnetization at zero field to that at 100K oe. That is, $A = B_r / 4\pi M_{100}$.

Particle size was determined by means of a Fisher Sub-Sieve Sizer.

The sintering furnace was an electrically heated ceramic tube.

All sintering was carried out in an inert atmosphere of purified argon and upon completion of the sintering, the sintered product was cooled in the same purified argon atmosphere.

Percent packing was determined from the measured density of the sample divided by the full density of the alloy under consideration. The full alloy densities used are as follows:

Alloy—	Gm./cm. ³
Co ₅ Sm	8.6
Co ₅ Ce _{0.5} Sm _{0.5}	8.4

B_s is the saturation induction.

B_r is the residual or remanent induction, i.e. the flux when the applied magnetic field is reduced to zero.

The intrinsic coercive force H_{ci} is the field strength at which the magnetization ($B-H$) or $4\pi M$ is zero.

Normal coercive force H_c is the field strength at which the induction B becomes zero.

The maximum energy product $(BH)_{max}$ represents the maximum product of the magnetic field H and the induction B determined on the demagnetization curve.

The term H_k helps characterize the squareness of the $4\pi M$ demagnetization curve. Specifically, H_k is the demagnetizing field required to drop the magnetization 10 percent below the remanence B_r . That is, $4\pi M_k = .9 B_r$, and H_k is the corresponding field strength. H_k is a useful parameter for evaluating demagnetization resistance.

EXAMPLE

In the runs of the following table, each alloy melt was made under purified argon by induction melting and cast into an ingot. The ingot was then crushed in air by means of mortar and pestle or in a jaw crusher in nitrogen and then ground in nitrogen by fluid energy milling into a powder of 6 to 8 microns average particle size. The sintering additive alloy was admixed with the base alloy by tumbling to form a substantially thorough mixture which was stable since the additive was substantially non-reactive in air and was slightly magnetic.

The green body of each run was formed by packing the mixture into a rubber tube having a working space of $\frac{3}{8}$ inch in diameter and $1\frac{1}{4}$ inches long. The tube was placed in an axial magnetic field of 60,000 to 100,000 oersteds to align the particles along the easy axis. After aligning, the tube was evacuated and the sample was hydrostatically pressed under 200,000 p.s.i. The pressed samples, i.e. green bodies, had a packing density of about 80 percent. The green bodies were cylindrical in form and had a diameter ranging from about $\frac{1}{4}$ up to about $\frac{3}{8}$ inch and a length ranging from about $\frac{3}{4}$ to $1\frac{1}{2}$ inches. Generally, the green bodies were machined to make a right cylinder of proper dimensions for testing purposes. The green bodies were sintered as indicated in the following table.

In the following table, the magnetic properties of the sintered products were determined after magnetization at room temperature in a field of 100,000 oersteds.

Run Number:	Base alloy						Sintering additive alloy						Green body			Sintering treatment		Sintered product		Magnetic properties of sintered product after magnetization					
	Percent			Weight (grams)			Percent			Weight (grams)			Ce per cent of Ce-Sn	Weight (grams)	Align-ment	Time (min.)	Temp. (° C.)	Den- sity (per- cent)	Pack- ing (per- cent)	B _s gauss	B _r gauss	H _k oersted	H _{ci} oersted	(BH) _{max} 10 ⁶ gauss oersted	
	Co	Sm	Ce	Co	Sm	Ce	Co	Sm	Ce	Co	Sm	Ce													
		Co	Sm	Ce	Co	Sm	Ce	Co	Sm	Ce	Co	Sm	Ce												
1	68.7	16.2	15.1	8.29	40.8	59.2	3.71	60.1	29.5	10.4	12	26.1	.89	30	1,100	7.73	92	6,640	5,400	3,600	1,300	6,300	6,300	5.5	
2	68.7	16.2	15.1	8.31	40.8	59.2	2.69	61.9	26.7	11.4	11.0	29.9	.95	30	1,100	8.24	97	8,340	7,650	3,800	1,300	6,300	4,000	13.5	
3	68.7	16.2	15.1	9.83	40	60	2.17	63.5	24.1	12.3	12.0	33.8	.94	60	1,075	7.5	88	8,700	7,200	3,800	1,300	6,300	10,800	12.8	
4	67.4	8.6	24.0	10.44	40	60	1.56	63.8	15.3	20.9	12.0	57.7	.92	60	1,075	7.9	93	7,900	6,700	3,800	1,300	6,300	4,900	10.7	
5	68.7	16.2	15.1	10.24	40	60	1.76	64.4	22.7	12.9	12.0	36.2	.91	60	1,075	7.1	84	9,200	7,000	3,600	1,300	4,700	4,700	11.0	
6	67.4	8.6	24.0	10.9	40	60	1.1	64.7	13.6	21.7	12.0	61.5	.94	60	1,075	8.0	94	8,800	7,700	3,800	1,300	4,000	4,000	12.7	
7	68.7	16.2	15.1	6.93	40	{ 30(Sm)	2.55	61	19.9	19.1	9.49	49.0	.909	30	1,100	-----	97.7	7,420	6,630	950	600	1,100	3.3		
8	68	32	0	9.8	40.8	{ 30(Ce)	2.2	63	37	0	12.0	0	.96	30	1,125	-----	94	9,330	8,450	7,500	7,200	19,500	17.6		

Run Nos. 1 through 7 illustrate the present product and permanent magnet as prepared by liquid phase sintering.

Although the table shows that the permanent magnet properties of the present magnets are somewhat lower than those of Run No. 8 where no cerium was used, the use of cerium in the present invention provides a number of advantages. It is a much more abundant element than samarium which makes it more practical to use. In addition, the permanent magnet properties of the present magnets makes permanent magnets available for a much wider variety of applications than has been possible heretofore.

The magnetic properties of the present sintered products can be improved by subjecting them to a heat-aging process to produce novel permanent magnets. This heat-aging process is substantially disclosed and claimed in copending U.S. patent application Ser. No. 33,315 entitled "Heat-Aged Sintered Cobalt-Rare Earth Intermetallic Product and Process," filed Apr. 30, 1970 in the names of Mark G. Benz and Donald L. Martin and assigned to the assignee hereof, and which by reference is made part of a disclosure of the present application.

As applied to the preparation of the new permanent magnets of the present invention, the heat-aging process comprises heating the present sintered products at a temperature within 400° C. below its sintering temperature for a period of time ranging up to 24 hours in a substantially inert atmosphere such as, for example, argon. The particular aging temperature and aging period is determinable empirically depending on the resulting improvement in magnetic properties produced. Specifically, such heat-aging of the present products usually increases their resistance to demagnetization as reflected by at least about a 15 percent increase in the H_k value of the resulting magnets. Generally, significant increases in the H_k factor are produced wherein the H_k factor is initially relatively low.

Copending U.S. patent application Ser. No. 33,224 entitled "Sintered Cobalt-Rare Earth Intermetallic Product," filed on Apr. 30, 1970, in the name of Mark G. Benz and assigned to the assignee hereof is, by reference, made part of the disclosure of the present application. In U.S. patent application Ser. No. 33,224, there is disclosed a process which, briefly stated, comprises forming a particulate alloy of cobalt and rare earth metal content substantially the same as that desired in the sintered product, compressing the particulate alloy into compact and sintering them to the desired density. The sintered product is comprised of a major amount of Co₅R intermetallic phase and up to about 35 percent by weight of the product of a second CoR phase which is richer in rare earth metal content than the Co₅R phase, where R is a rare earth metal. These sintered products are then magnetized to form novel permanent magnets.

In copending U.S. patent application Ser. No. 66,173 entitled "Sintered Cobalt-Rare Earth Intermetallic Product and Permanent Magnets Produced Therefrom" filed of even date herewith in the name of Donald L. Martin and assigned to the assignee hereof, there is disclosed novel sintered products comprised of intermetallic compounds of samarium and praseodymium and permanent magnets produced therefrom.

In copending U.S. patent application Ser. No. 66,173 entitled "Sintered Cobalt-Rare Earth Intermetallic Product Including Samarium and Lanthanum and Permanent Magnets Produced Therefrom" filed of even date herewith in the name of Donald L. Martin and assigned to the assignee hereof, there is disclosed novel sintered products comprised of intermetallic compounds of cobalt and rare earth metals composed of samarium and lanthanum and permanent magnets produced therefrom.

In copending U.S. patent application Ser. No. 66,216 entitled "Sintered Intermetallic Product of Cobalt, Samarium and Cerium Mischmetal and Permanent Magnets Produced Therefrom" filed of even date herewith

in the names of Donald L. Martin and Mark G. Benz and assigned to the assignee hereof, there is disclosed novel sintered products comprised of intermetallic compounds of cobalt and rare earth metals composed of samarium and cerium mischmetal and permanent magnets produced therefrom.

All of the above cited patent applications are, by reference, made part of the disclosure of the present application.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. A permanent magnet having substantially stable permanent magnet properties and having as the active magnetic component a sintered product of compacted particulate cobalt-rare earth intermetallic material, said sintered product having pores which are substantially non-interconnecting, a packing of at least 87 percent and a composition consisting essentially of cobalt in an amount of 60 to 66 percent by weight of said sintered product and the rare earth metals of samarium and cerium in an amount of 34 to 40 percent by weight of said sintered product with said cerium ranging in amount from about 10 to 90 percent by weight of the total rare earth metals content.

2. A permanent magnet according to claim 1 wherein said cerium is present in an amount ranging from about 26.1 to 61.5 percent by weight of the total rare earth metals content.

3. A permanent magnet having substantially stable permanent magnet properties having as the active magnetic component particles of a sintered product of compacted particulate cobalt-rare earth intermetallic material, said particles of said sintered product being bonded to a matrix material, said sintered product having pores which are substantially non-interconnecting, a packing of at least 87 percent and a composition consisting essentially of

cobalt in an amount of 60 to 66 percent by weight of said sintered product and the rare earth metals of samarium and cerium in an amount of 34 to 40 percent by weight of said sintered product with said cerium ranging in amount from about 10 to 90 percent by weight of the total rare earth metals content.

4. A permanent magnet according to claim 3 wherein said matrix material is a metal.

5. A permanent magnet according to claim 3 wherein said matrix material is a plastic.

6. A permanent magnet according to claim 3 wherein said matrix material is a rubber.

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U.S. Cl. X.R.

75—170; 148—101