

- [54] **PERMANENT MAGNETS OF COBALT, SAMARIUM, GADOLINIUM ALLOY**
- [75] Inventors: **Mark G. Benz**, Burnt Hills; **Donald L. Martin**, Elnora, both of N.Y.
- [73] Assignee: **General Electric Company**, Schenectady, N.Y.
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- [51] Int. Cl.<sup>2</sup> ..... **H01F 1/04**
- [58] Field of Search ..... **148/31.57, 101, 103, 105; 75/84, 152**

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*Primary Examiner*—Walter R. Satterfield

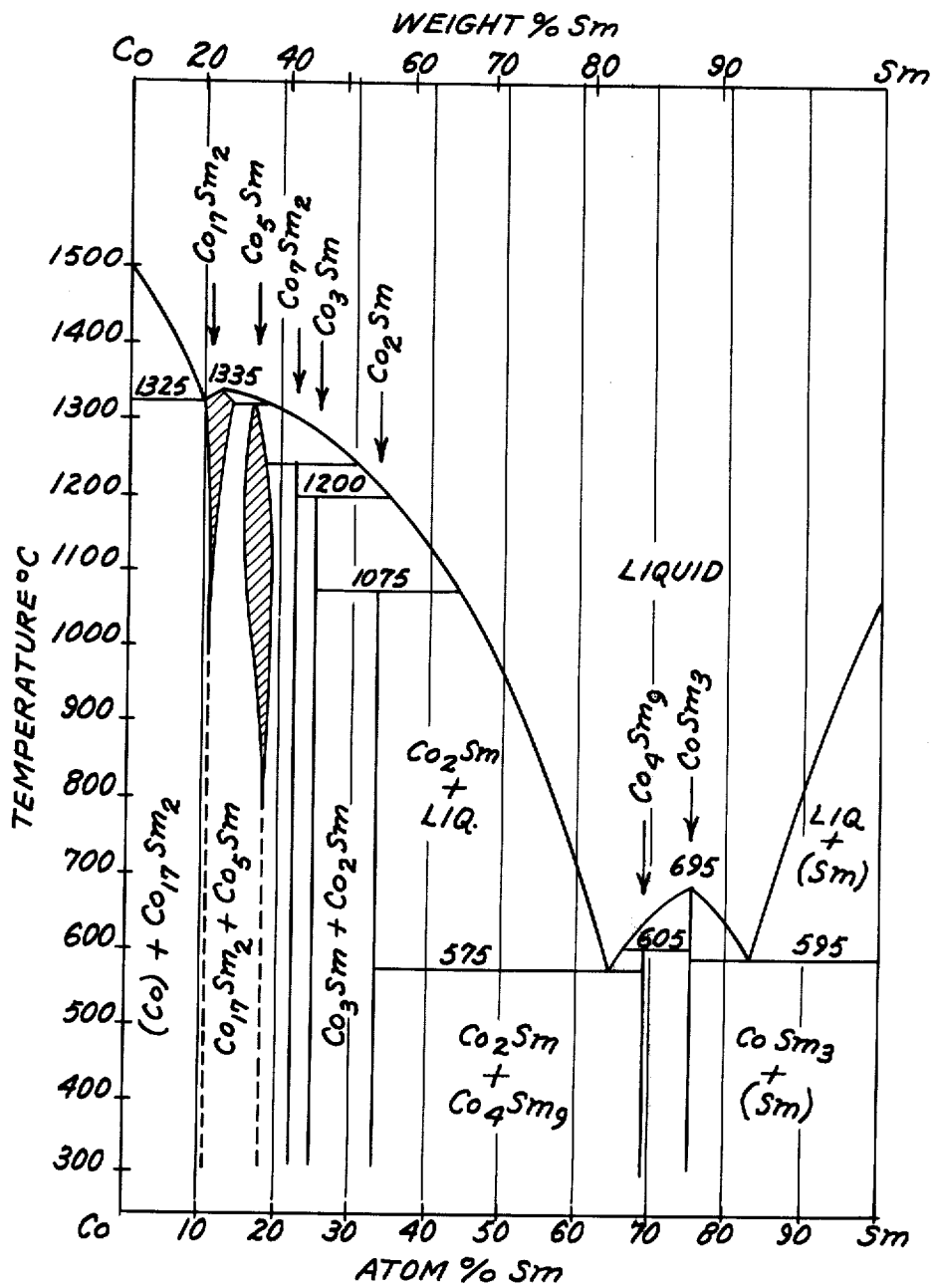
*Attorney, Agent, or Firm*—Jane M. Binkowski; Joseph T. Cohen; Jerome C. Squillaro

[57] **ABSTRACT**

A permanent magnet having as the active magnetic component a sintered product consisting essentially of cobalt-samarium-gadolinium alloy having a reversible magnetization temperature coefficient in air ranging from -0.030% per °C to +0.015% per °C over a temperature range of about -50°C to +300°C, a saturation magnetization  $4\pi J_s$  of at least 5 kilogauss and a coercive force  $H_c$  of at least -5,000 oersteds.

**1 Claim, 4 Drawing Figures**

Fig. 1.



SHEET 2 OF 4

Fig. 2.

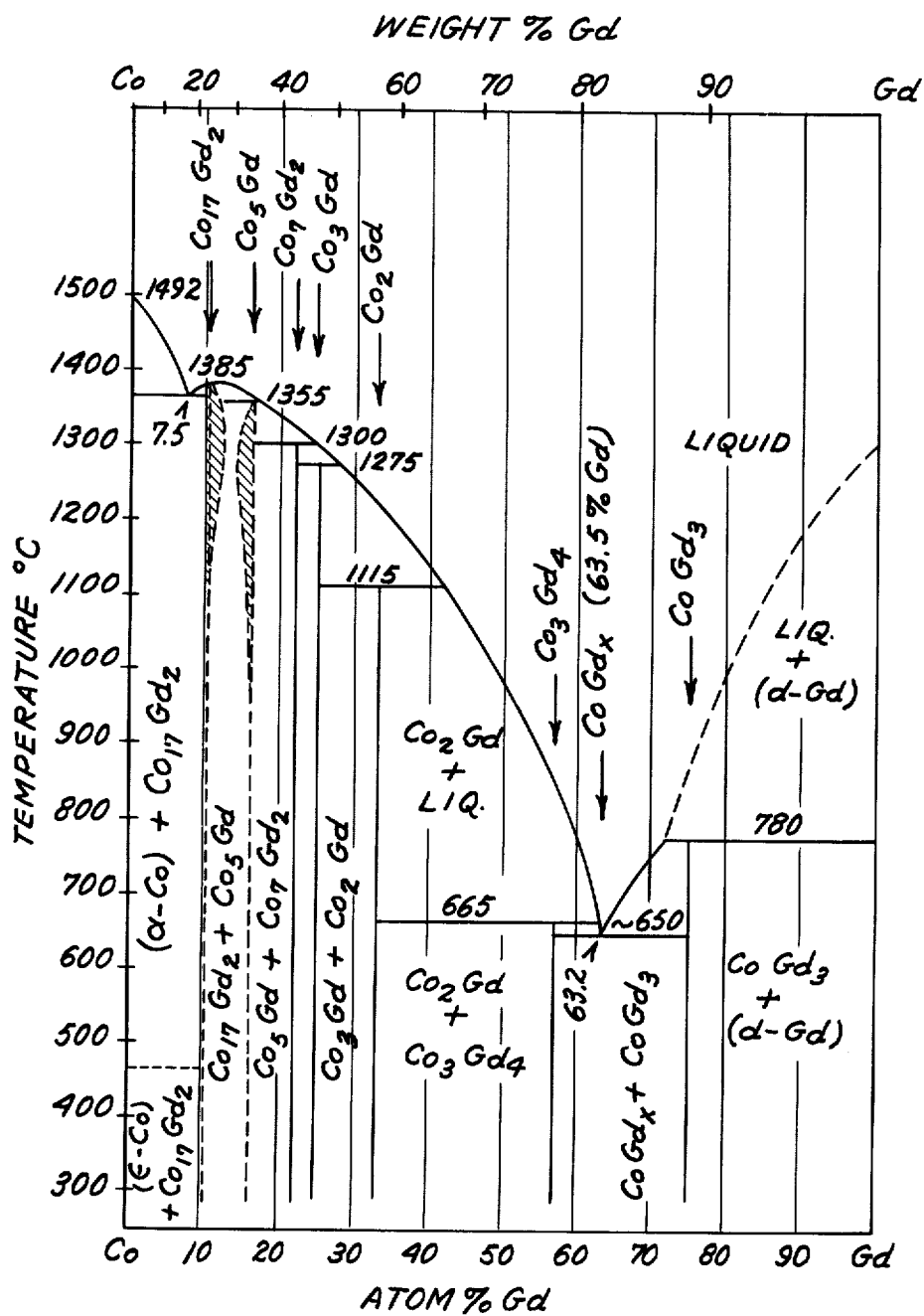
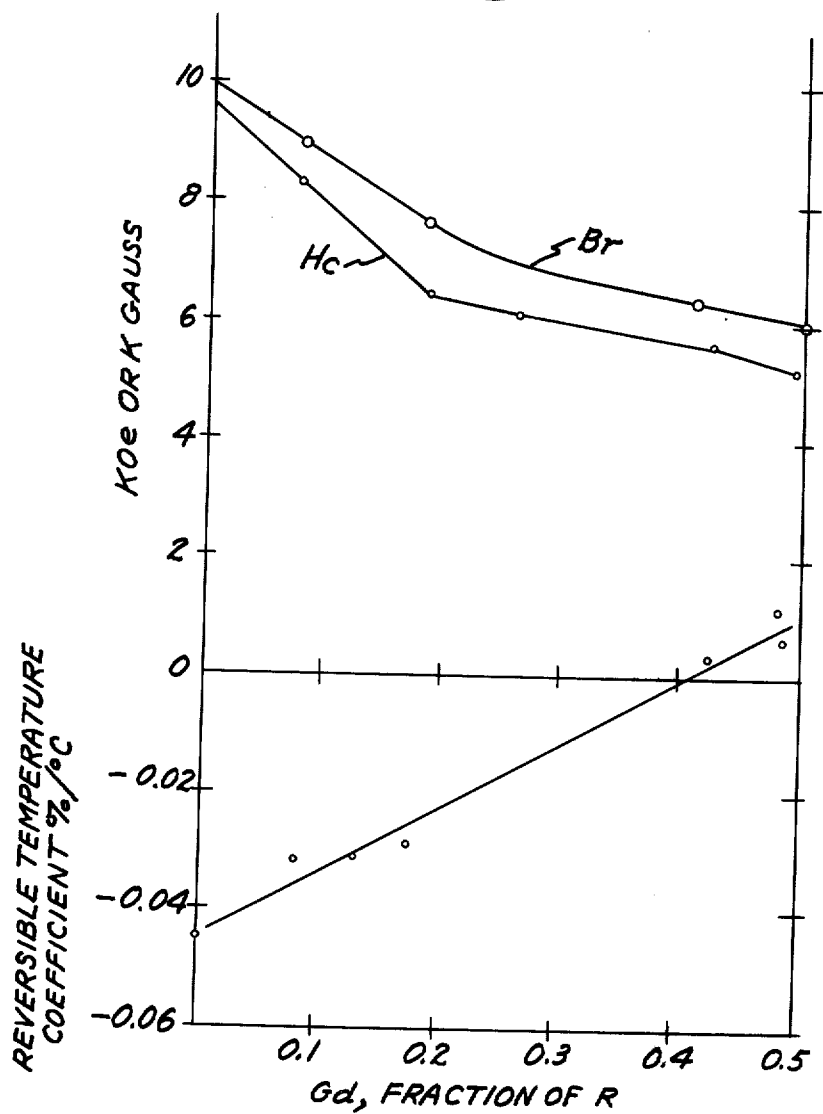
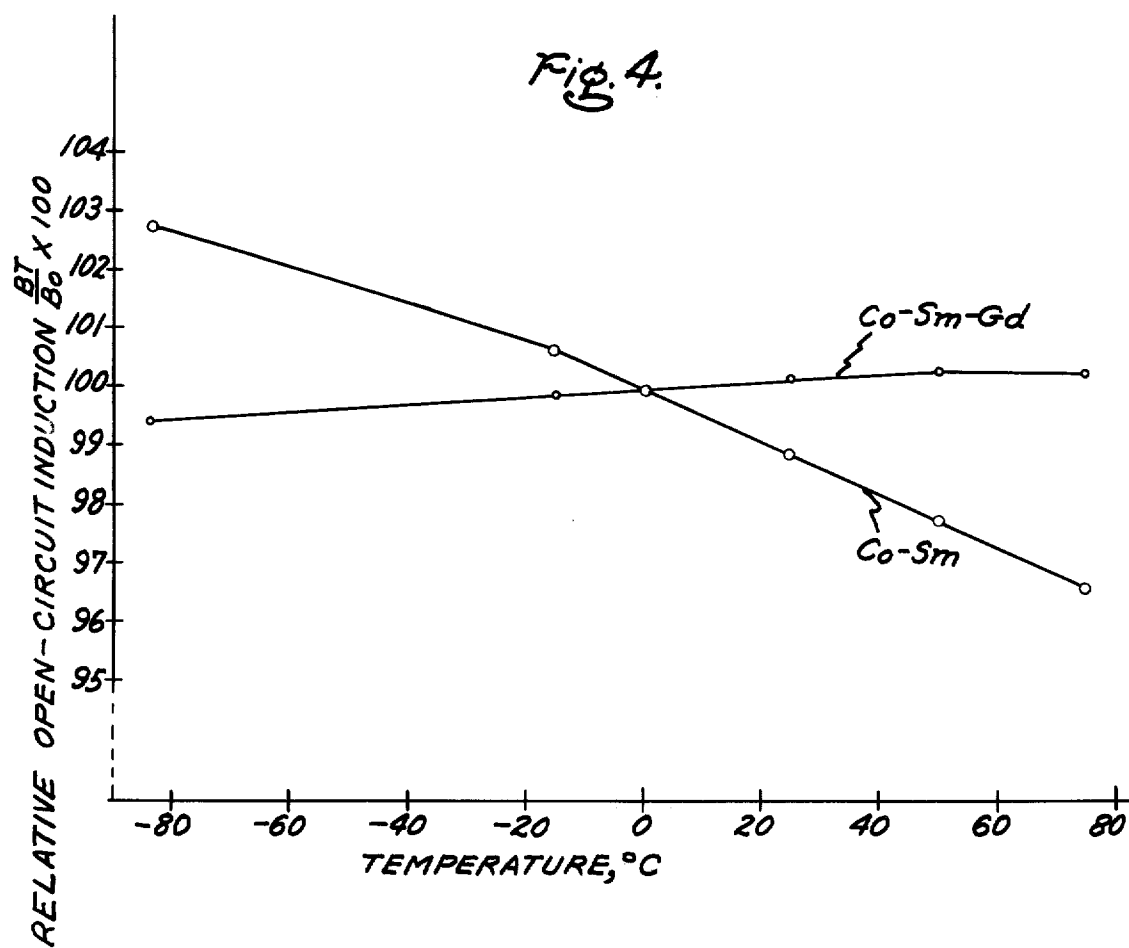


Fig. 3.





## PERMANENT MAGNETS OF COBALT, SAMARIUM, GADOLINIUM ALLOY

The present invention relates generally to the permanent magnet art and is more particularly concerned with novel permanent magnets of cobalt, samarium, and gadolinium having useful permanent magnet properties and a reversible magnetization temperature coefficient which is significantly low, zero or positive within a broad temperature range.

Within the past few years a new class of materials for making permanent magnets has been developed, based on cobalt and rare-earth elements. The improvement over prior art materials is so great that the cobalt-rare-earth magnets stand in a class by themselves. In terms of their resistance to demagnetization the new materials are from 20 to 50 times superior to conventional magnets of the Alnico type, and their magnetic energy is from two to six times greater. Since the more powerful the magnet for a given size is the smaller it can be for a given job, the cobalt-rare-earth magnets have applications for which prior art materials cannot even be considered.

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 figures accompanying and forming a part of the specification, in which:

FIG. 1 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.

FIG. 2 is the cobalt-gadolinium phase diagram.

FIG. 3 is a chart bearing curves which illustrate how the substitution of increasing amounts of gadolinium for samarium affects the properties of the resulting ternary alloy magnet. Specifically, the reversible temperature coefficient was measured for the range 20°C to 225°C, and the remanent induction  $B_r$ , and coercive force  $H_c$  were measured at room temperature, 20°C.

FIG. 4 is a chart bearing curves comparing the change in open circuit induction  $B_o$  over a broad temperature range of a cobalt-samarium permanent magnet with a cobalt-gadolinium-samarium magnet prepared in substantially the same manner.

When a permanent magnet material is magnetized, a magnetization value of  $4\pi J$  gauss is established therein. The shape of the magnet or the magnetic circuit impose a demagnetizing field of  $H$  oersteds. Together, these properties,  $4\pi J$  and  $H$  equal the flux density  $B$  which is also measured in gauss.

Probably the most desirable property of a permanent magnet is that it provide useful external magnetic energy which is constant. This is important because in practically all applications the permanent magnet must function under a variety of demagnetizing influences. Specifically, changes in magnetization with temperature are of concern to the engineer designing permanent magnet devices. Of particular interest is the reversible loss in magnetization or flux which occurs when a magnet is heated to an elevated temperature. It would be desirable for a permanent magnet to undergo no reversible loss in magnetization or flux in the operating temperature range. For example, wattmeters now generally contain conventional magnets which require highly complex circuitry to compensate for changes in magnetization or flux due to changes in temperature

and such circuitry adds significantly to the weight and mass of the product.

As used herein, the reversible loss is the change in saturation magnetization  $4\pi J_s$  measured at the elevated temperature compared to that measured at room temperature.

Permanent magnets of cobalt and samarium, particularly those magnets containing a substantial amount of the  $\text{Co}_5\text{Sm}$  phase, appear to have the most desirable magnetic properties. However, as illustrated by FIG. 3, the permanent magnet of cobalt and samarium has a reversible magnetization temperature coefficient of  $-0.045\%$  per °C. Such a loss in magnetization may not appear to be significant but over a range of 100° it undergoes a loss of magnetization of 4.5 percent which is significant for a number of applications such as the aforementioned wattmeters.

Heretofore, permanent magnets having a suitable combination of magnetic properties and reversible magnetization temperature coefficient over a desirable broad temperature range have not been produced. In accordance with the present invention, such a magnet is produced and it consists essentially of a sintered product of a ternary metal alloy of cobalt, gadolinium and samarium of particular composition.

Briefly stated, the present invention is comprised of a permanent magnet wherein the active magnetic component consists essentially of a sintered product consisting essentially of compacted particulate ternary metal alloy of cobalt and a rare earth component. The rare earth component is present in an amount ranging from 16.6 atomic % to 20 atomic % based on the ternary metal alloy and consists essentially of samarium and gadolinium with the gadolinium atom fraction ranging from 0.12 to less than 0.5 of the rare earth content of the  $\text{Co-Sm-Gd}$  alloy. The sintered product consists essentially of at least two phases with one of the phases being a  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  alloy phase and the second of said phases being a  $\text{Co-Gd-Sm}$  alloy phase richer in rare earth content than the  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  alloy phase. The sintered product has a density of at least 87 percent of theoretical and has pores which are substantially noninterconnecting. The present permanent magnet has a saturation magnetization  $4\pi J_s$  of at least 5 kilogauss, a coercive force  $H_c$  of at least  $-5,000$  oersteds and a reversible magnetization temperature coefficient in air ranging from  $-0.030\%$  per °C to  $+0.015\%$  per °C over a temperature range of about  $-50^\circ\text{C}$  to  $+300^\circ\text{C}$ .

As a result of the present invention, permanent magnets can be prepared having a predetermined reversible temperature coefficient.

In the present invention the reversible magnetization temperature coefficient % per °C is calculated in accordance with the following equation:

$$\frac{4\pi J_s - 4\pi J_{20}}{4\pi J_{20} (t - 20^\circ\text{C})} \times 100 = \text{reversible temp. coefficient \% per } ^\circ\text{C}.$$

Specifically, the sintered product is initially magnetized at room temperature to saturation magnetization,  $4\pi J_s$ . The resulting magnet is then heated or cooled to the temperature of interest and the magnetization measured at that temperature of interest,  $4\pi J_t$ , and then the magnetization is measured at room temperature,  $4\pi J_{20}$ .

As used herein, the coercive force,  $H_c$ , is the demagnetizing field necessary to reduce the induction,  $B$ , to zero.

The present magnets have a wide variety of applications. They are highly useful in wattmeters where their particular properties eliminate the need for means which compensate for reversible changes in magnetization or flux due to temperature change. Also, the present magnet with a positive reversible magnetization temperature coefficient may be particularly preferred since in such instance the magnetization or flux would increase with increasing temperature and could be used as a means to compensate for changes in circuitry due to temperature change. In addition, products utilizing the present magnets can be redesigned to have smaller housings and have significantly lower weight.

The present sintered product consists essentially of a ternary metal alloy of cobalt and a rare earth component composed of gadolinium and samarium which is present in an amount ranging from 16.6 atomic % to 20 atomic % based on the ternary metal alloy. Should the rare earth content be less than 16.6 atomic % of the ternary metal alloy, the sintered product would consist of a single phase  $\text{Co}_5(\text{GdSm})$  alloy or phases containing less rare earth with properties significantly inferior to those of the present magnet. Also, should the ternary metal alloy contain rare earth in excess of 20 atomic %, its properties likewise would be significantly inferior to that of the present magnet.

In addition, to produce the present magnet, the gadolinium atom fraction of the rare earth content of the Co-Sm-Gd metal alloy must range from 0.12 to less than 0.5. The particular amount of gadolinium used will depend upon the desired saturation magnetization level, the reversible change with temperature being sought, and the temperature range of interest. Amounts of gadolinium less than the 0.12 atom fraction of the rare earth content do not produce a significant change in the reversible magnetization temperature coefficient per  $^{\circ}\text{C}$  and atom fractions of gadolinium of 0.5 or higher of the rare earth content of the metal alloy significantly deteriorate permanent magnet properties, e.g., the resulting magnet would not have a saturation magnetization  $4\pi J_s$  of at least 5 kilogauss and a coercive force  $H_c$  of at least -5000 oersteds.

The present sintered product consists essentially of at least two phases with one of said phases being a  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  alloy phase and the second of said phases being a Co-Gd-Sm phase richer in rare earth content than the  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  alloy phase.

The present sintered product is produced only in accordance with the methods disclosed in U.S. Pat. Nos. 3,655,464, 3,655,463, 3,695,945, and 3,684,593, all of which by reference are made part of the disclosure of the present application.

Each of the aforementioned patents discloses a process for preparing novel sintered cobalt-rare earth intermetallic products which can be magnetized to form permanent magnets having improved magnetic properties.

Briefly stated, to prepare the present sintered ternary alloy product in accordance with the disclosure of U.S. Pat. No. 3,655,464 a compact of a particulate mixture of a base Co-R alloy which can be Co-Sm, Co-Gd or Co-Gd-Sm and an additive Co-R alloy which likewise can be Co-Sm, Co-Gd or Co-Gd-Sm is sintered to produce a product consisting essentially of at least two

phases with one of the phases being a  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  alloy phase and the second of the phases being a  $\text{Co}_7(\text{Gd}_y\text{Sm}_{2-y})$  alloy phase. Specifically, the base alloy is one which at sintering temperatures exists as a solid  $\text{Co}_5\text{R}$  intermetallic single phase. 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. The 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.

The base and additive alloys, in particulate form, are each used in an amount to form a mixture which has a cobalt, gadolinium and samarium content substantially corresponding to that required in the final desired sintered product. The additive alloy should be used in an amount sufficient to promote sintering, and generally, should be used in an amount of at least 0.5 percent by weight of the base-additive alloy mixture. The particulate mixture is compressed into a green body of the desired size and density. Preferably, the particles are magnetically aligned along their easy axis prior to or during compression since the greater their magnetic alignment, the better are the resulting magnetic properties.

The green body is sintered in a substantially inert atmosphere to produce a sintered body of desired density wherein the pores are substantially non-interconnecting, which generally is a sintered body having a density of at least about 87 percent of theoretical. 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.

Sintering temperature depends largely on the particular cobalt, samarium, gadolinium composition being sintered and generally ranges from about  $1,075^{\circ}\text{C}$  to about  $1,200^{\circ}\text{C}$  with a sintering temperature of  $1,100^{\circ}\text{C}$  usually being particularly satisfactory.

The procedure for forming sintered products disclosed in U.S. Pat. No. 3,655,463 is substantially the same as that disclosed in U.S. Pat. No. 3,655,464 except that an additive Co-R alloy which is solid at sintering temperature and which is richer in rare earth metal than the base alloy is used.

The procedure for forming the sintered products disclosed in U.S. Pat. No. 3,695,945 is substantially the same as that disclosed in U.S. Pat. No. 3,655,464 except that a cobalt, gadolinium, samarium alloy of proper composition is initially formed.

In U.S. Pat. No. 3,684,593 there is disclosed a process for preparing heat-aged novel sintered cobalt-rare earth intermetallic products by providing a sintered cobalt-rare earth intermetallic product ranging in composition from a single solid  $\text{Co}_5\text{R}$  phase to that composed of  $\text{Co}_5\text{R}$  phase and a second phase of solid Co-R in an amount of up to about 30 percent by weight of the product and richer in rare earth metal content than said  $\text{Co}_5\text{R}$ , and heat-aging said product at an aging temperature within  $400^{\circ}\text{C}$  below the temperature at which it was sintered to precipitate Co-R phase richer in rare earth metal content than said  $\text{Co}_5\text{R}$  in an amount sufficient to increase intrinsic and/or normal coercive force of said product by at least 10 percent. This particular single phase product is of a composition at sintering temperature close to the boundary or solution line defining the single solid  $\text{Co}_5\text{R}$  phase on the rare earth rich side. Heat-aging is carried out in an atmosphere such

as argon in which the material is substantially inert. The precipitated Co-R phase is generally present in an amount ranging from about 1 to 15 percent by weight of the product. In the present invention, the precipitated phase is a Co-Gd-Sm phase richer in rare earth content than the  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  alloy phase. These heat-aged sintered products are particularly useful in the present invention.

netization  $4\pi J_s$  at room temperature along its easy axis of magnetization in a field of 60 kiloersteds. The properties of each resulting magnet were determined and are shown in Table I where Run No. 1 is a control and where Run Nos. 3-6 illustrate the present invention.

In Table I the Reversible Temperature Coefficient % per °C was determined over the temperature range 20°C to 225°C.

TABLE I

| Run No. | Gd + Sm Atomic % | Gd Fraction of Gd + Sm | Saturation Magnetization $4\pi J_s$ Kilogauss | Energy Product (BH) max. ( $10^6$ gauss $\times$ oersteds) | Reversible Temperature Coefficient % per °C | Coercive Force $H_c$ Kiloersteds | Remanent Induction $B_r$ Kilogauss |
|---------|------------------|------------------------|-----------------------------------------------|------------------------------------------------------------|---------------------------------------------|----------------------------------|------------------------------------|
| 1       | 16.7             | 0                      | 10.9                                          | 23.4                                                       | -.04                                        | -9.5                             | 9.7                                |
| 2       | 16.8             | .08                    | 9.7                                           | 20.1                                                       | -.032                                       | -8.20                            | 9.0                                |
| 3       | 16.7             | .18                    | 8.8                                           | 12.3                                                       | -.028                                       | -6.50                            | 7.6                                |
| 4       | 16.8             | .42                    | 7.1                                           | 9.3                                                        | +.004                                       | -5.7                             | 6.4                                |
| 5       | 17.0             | .48                    | 6.7                                           | 7.6                                                        | +.012                                       | -5.3                             | 5.6                                |
| 6       | 16.9             | .49                    | 7.0                                           | 9.1                                                        | +.007                                       | -6.0                             | 6.1                                |

The present sintered product is magnetized along the easy or "C" axis of magnetization preferably to full saturation magnetization or to at least approach saturation magnetization, i.e. within about 10 percent of full saturation magnetization. The present permanent magnet has a wide variety of applications and is particularly useful in wattmeters, computers, and microwave devices.

If desired, the sintered bulk product of the present invention can be crushed to a desired particle size and bonded to a non-magnetic matrix material to produce the present permanent magnet. The non-magnetic matrix material may vary widely and may be plastic, rubber or metal such as, for example lead, tin, zinc, copper or aluminum.

The invention is further illustrated by the following examples.

## EXAMPLE 1

Alloy compositions containing between 16 atomic % and 37 atomic % Sm and/or Gd were prepared by induction melting and chill casting. Each alloy was reduced to a fine powder of a particle size between 5-10 microns by a series of steps which included jaw crushing, double disk pulverizing, and milling in a fluid energy mill with nitrogen as the working gas. Several Co-Sm and Co-Sm-Gd alloy powders were blended together in the appropriate ratio required to achieve the desired final average composition in each blend. Also, each blend contained at least one powder which functioned as the solid or liquid sintering additive as set forth in U.S. Pat. Nos. 3,655,464 and 3,655,463.

For each run in Table I, the powder blend was oriented in a magnetic field of 60 kiloersteds along its C or easy axis of magnetization. A low pressure was applied to the oriented sample in order to retain the alignment while the sample was transferred to the high pressure chamber. By application of a hydrostatic pressure of approximately 13.6 kbars, the oriented sample was compacted to a relative density of 80 percent. The resulting compact was sintered in an atmosphere of argon at a temperature of approximately 1,120°C for about one hour to densities ranging between 90 and 95 percent of theoretical. The sintered compact was then furnace cooled to temperatures ranging from 800°C to 1,050°C at which temperature it was heat-aged for ½ to 25 hours. Each resulting sintered product was about the same size and was magnetized to saturation mag-

Table I illustrates how the substitution of increasing amounts of gadolinium for samarium affects the properties of the present resulting magnets. Specifically, Run Nos. 3-6, which illustrate the present invention, show permanent magnets with very low or positive reversible temperature coefficients having a saturation magnetization of at least 5 kilogauss, a coercive force of at least -5 kiloersteds, and high energy products.

Some of the properties of Table I are shown graphically in FIG. 3 which shows that a gadolinium fraction of 0.12 is necessary to achieve a reversible magnetization temperature coefficient of -0.030% per °C.

Metallographic examination of Run Nos. 3, 4 and 5 showed them to consist essentially of two phases, e.g. a  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  phase and a Co-Gd-Sm phase richer in rare earth metal content than the  $\text{Co}_5(\text{Gd}_x\text{Sm}_{1-x})$  phase. The samples also contained minor amounts of an oxide phase consisting of  $\text{Sm}_2\text{O}_3$  and  $\text{Gd}_2\text{O}_3$ .

## EXAMPLE 2

In this example a Co-Gd-Sm permanent magnet illustrating the present invention was prepared and its properties compared to a Co-Sm permanent magnet prepared in substantially the same manner but containing no gadolinium.

Specifically, to prepare the Co-Gd-Sm permanent magnet, three powders having an average particle size of about 6 microns were blended together to make the final blend which was then magnetically aligned along the easy axis of magnetization and hydropressed to form a green body. The composition of each powder and the blend % of each was as follows:

| Powder No. | Wt. % in Blend | Wt. % Co | Wt. % $\text{O}_2$ | Wt. % Sm | Wt. % Gd |
|------------|----------------|----------|--------------------|----------|----------|
| A          | 42.6           | 65.7     | 0.32               | 17.0     | 17.0     |
| B          | 13.8           | 65.5     | 0.42               | 34.1     | —        |
| C          | 43.6           | 58.9     | 0.95               | 20.1     | 20.1     |

The composition of the final blend was 62.7 wt. % Co, 0.61 wt. %  $\text{O}_2$ , 20.7 wt. % Sm, and 16.0 wt. % Gd. Assuming the oxygen to be combined with equal amounts of Sm and Gd, the atomic composition of the alloy was calculated to be: 83.3 A/o, Co, 9.8 A/o Sm and 7.0 A/o Gd. The Gd fraction in the metallic phase was 0.416 or 41.6 atomic % of the total rare earth component.

The green body was sintered in an atmosphere of argon at a temperature of 1,140°C for 1 hour and then furnace cooled to 925°C at which temperature it was heat-aged for 65 hours and then cooled to room temperature.

The resulting sintered body was magnetized at room temperature in air along its easy axis of magnetization in a magnetizing field of 60,000 oersteds. The magnetic properties of the resulting magnet were measured in air at 25°C and then at 225°C and the results are shown in Table II.

TABLE II

| Magnetic Properties                                                        | 25°C          | 225°C         |
|----------------------------------------------------------------------------|---------------|---------------|
| Saturation magnetization, $4\pi J_s$                                       | 7.05 K Gauss  | 7.10 K Gauss  |
| Remanent induction, $B_r$                                                  | 6.35 K Gauss  | 6.30 K Gauss  |
| Coercive force, $H_c$                                                      | -5.67 K Oers. | -5.16 K Oers. |
| Energy product<br>( $BH$ ) <sub>m</sub> (10 <sup>6</sup> gauss × oersteds) | 9.3 MG Oers.  | 8.0 MG Oers.  |

Table II illustrates that critical magnetic properties such as saturation magnetization, remanent induction and coercive force of the Co-Gd-Sm permanent magnet are substantially constant over temperatures ranging from 25°C to 225°C.

The open circuit induction  $B_o$  of the Co-Gd-Sm magnet was determined to be 6.15 kilogauss at room temperature. The Co-Gd-Sm magnet was then cooled to -80°C and its open circuit induction at various temperatures  $B_o$  ranging from -80°C to +80°C was determined. The results are graphically illustrated in FIG. 4 which shows a slight positive slope for the Co-Gd-Sm magnet over the range -75°C to +75°C. The reversible magnetization temperature coefficient of the Co-Gd-Sm magnet was determined to be +0.004% per °C which indicates that its magnetization  $4\pi J$  increases with increasing temperature. The Co-Gd-Sm magnet had a density of 92.3 percent of theoretical and its pores were substantially non-interconnecting.

As a control, a Co-Sm permanent heat-aged was prepared in substantially the same manner as the Co-Gd-Sm permanent magnet. The nominal composition of the final Co-Sm powder blend was 62.75 wt.% Co and 37.25 wt.% Sm. A green body of the Co-Sm blend powder was formed in the same manner and sintered at a temperature 1,115°C for 1 hour, then furnace cooled to 875°C at which temperature it was heataged for ½ hour and then cooled to room temperature. It was then

magnetized in the same manner.

The resulting Co-Sm permanent magnet had an open circuit induction  $B_o$  at room temperature of 8.26 kilogauss. The open circuit induction of the Co-Sm magnet was then measured at temperatures ranging from -80°C to +80°C as illustrated in FIG. 4. FIG. 4 shows that the open circuit induction of the Co-Sm permanent magnet decreases significantly at temperatures ranging from 0°C to +75°C. The reversible magnetization temperature coefficient of the Co-Sm magnet was determined to be -0.045% per °C.

What is claimed is:

1. A permanent magnet with substantially stable permanent magnet properties and characterized by significantly low or zero reversible magnetization temperature coefficient in air through a wide temperature range consisting essentially of a sintered product of compacted particulate ternary metal alloy consisting essentially of cobalt and rare earth component, said rare earth component ranging in amount from 16.6 atomic % to 20 atomic % of said ternary metal alloy and consisting essentially of samarium and gadolinium with the gadolinium atom fraction ranging from 0.12 to less than 0.5 of said rare earth component of said ternary alloy, said alloy consisting essentially of two phases with one of said phases being a  $\text{Co}_5\text{GdSm}$  alloy phase and the second of said phases being a  $\text{CoGdSm}$  alloy phase richer in rare earth content than said  $\text{Co}_5\text{GdSm}$  alloy phase, said sintered product having a density of at least 87 percent of theoretical and having pores which are substantially non-interconnecting, and said permanent magnet having a reversible magnetization temperature coefficient in air ranging from -0.030% per °C to +0.015% per °C over the temperature range of -50°C to +300°C, a saturation magnetization  $4\pi J_s$  of at least 5 kilogauss and a coercive force  $H_c$  of at least -5,000 oersteds in said temperature range.

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