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Benz

[11] **3,844,850**

[45] **Oct. 29, 1974**

[54] **LARGE GRAIN COBALT-SAMARIUM INTERMETALLIC PERMANENT MAGNET MATERIAL AND PROCESS**

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[21] Appl. No.: **244,424**

[52] **U.S. Cl.** **148/103**, 148/31.57, 148/102, 148/101

[51] **Int. Cl.** **H01f 1/02**

[58] **Field of Search** 148/103, 102, 101, 100, 148/105, 31.57; 75/170

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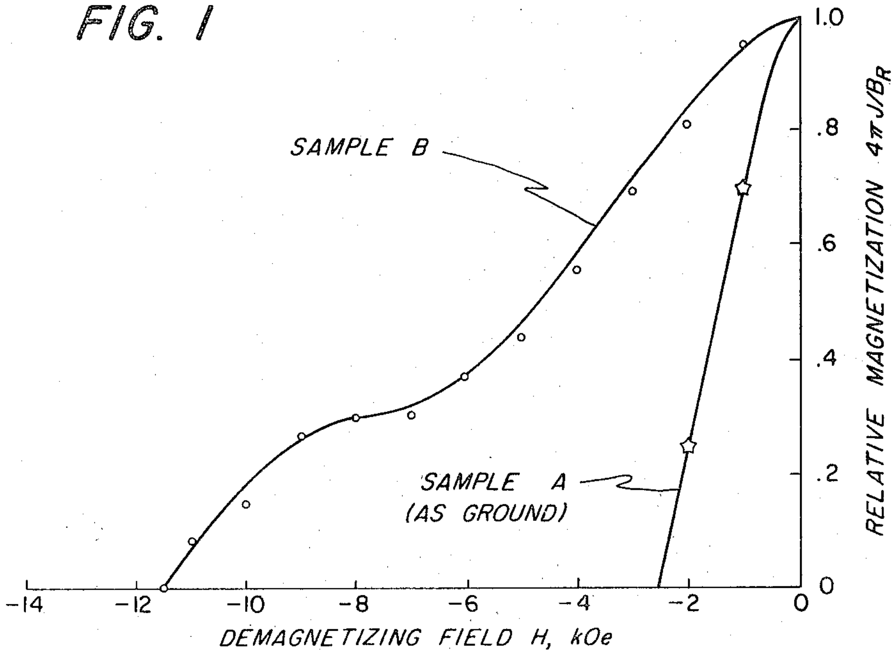
[57] **ABSTRACT**

An alloy of cobalt and samarium is cast to produce a cast body having grains ranging in size from about 100 microns to 1000 microns. The cast body is annealed in an atmosphere in which it is substantially inert at a temperature ranging from about 900°C up to a temperature below its melting point for a period of time ranging from about 5 minutes to 24 hours. To get significantly useful permanent magnet properties, it should be annealed at a particular annealing temperature for a period of time sufficient for the resulting free grains to show at room temperature, after being magnetized to at least approach saturation magnetization, a relative magnetization value of $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds with relative magnetization $4\pi J/B_r$, by definition, being 1.00 at zero demagnetizing field. The annealed body is then comminuted to a grain size corresponding to or smaller than the grain size of the cast body and ranging from about 50 microns to 200 microns.

13 Claims, 4 Drawing Figures

FIG. 1


SAMPLE B
SAMPLE A
(AS GROUND)
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.6
.4
.2
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RELATIVE MAGNETIZATION $4\pi J/B_R$
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-12
-10
-8
-6
-4
-2
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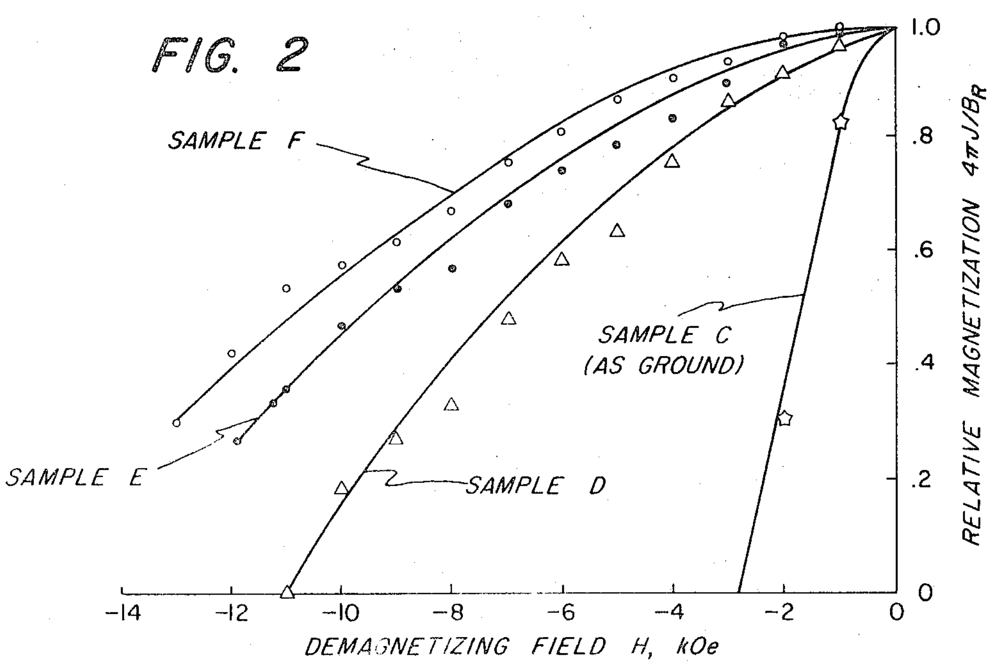
DEMAGNETIZING FIELD H, kOe

FIG. 2


SAMPLE F
SAMPLE E
SAMPLE D
SAMPLE C
(AS GROUND)
1.0
.8
.6
.4
.2
0
RELATIVE MAGNETIZATION $4\pi J/B_R$
-14
-12
-10
-8
-6
-4
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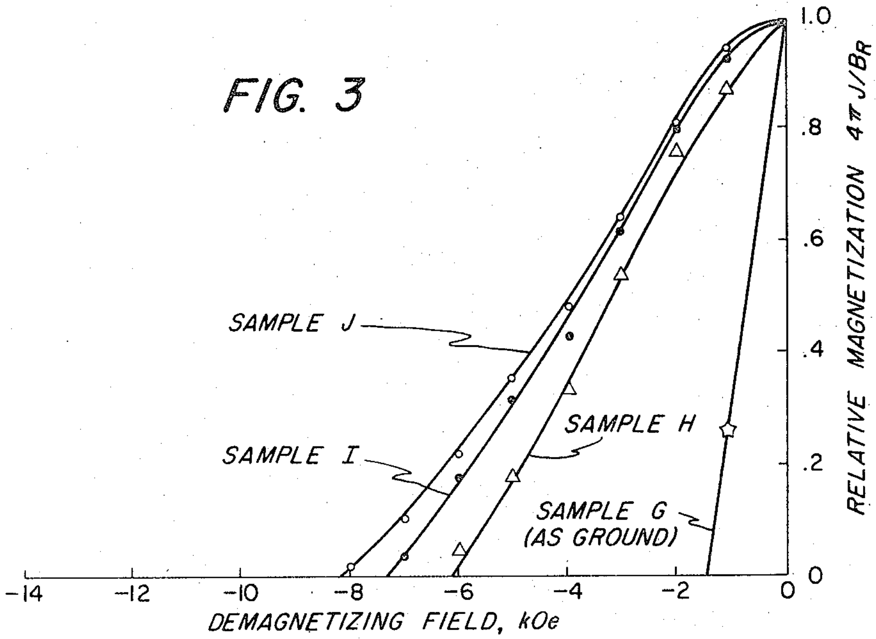
DEMAGNETIZING FIELD H, kOe

FIG. 3


SAMPLE J
SAMPLE I
SAMPLE H
SAMPLE G
(AS GROUND)
1.0
.8
.6
.4
.2
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RELATIVE MAGNETIZATION 4π J/BR
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-12
-10
-8
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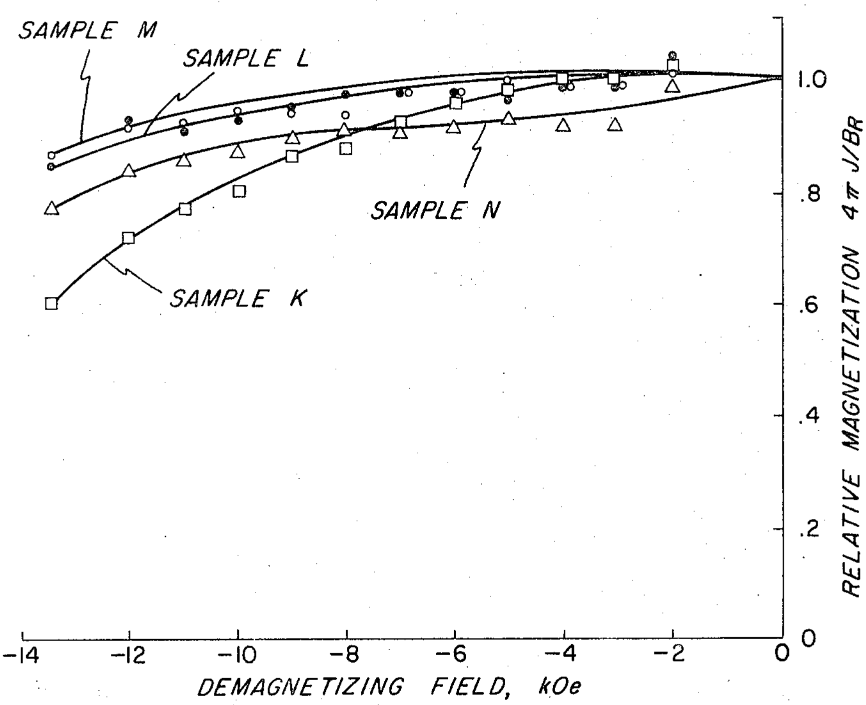
-4
-2
0
DEMAGNETIZING FIELD, kOe

FIG. 4


SAMPLE M
SAMPLE L
SAMPLE N
SAMPLE K
1.0
.8
.6
.4
.2
0
RELATIVE MAGNETIZATION 4π J/BR
-14
-12
-10
-8
-6
-4
-2
0
DEMAGNETIZING FIELD, kOe

LARGE GRAIN COBALT-SAMARIUM INTERMETALLIC PERMANENT MAGNET MATERIAL AND PROCESS

The present invention relates generally to the art of permanent magnets. In one aspect, it is concerned with making novel granular cobalt-samarium intermetallic permanent magnet material having unique characteristics. In another aspect it is concerned with permanent magnets comprised of this novel granular magnetic material bonded to a non-magnetic matrix.

Permanent magnets, i.e., "hard" magnetic materials, 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.

Within the past few years a new clas of materials for making permanent magnets has been developed based on cobalt and rare-earth elements, particularly cobalt and samarium. 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 superior to conventional magnets of the Alnico and ferrite type, and their magnetic energy is significantly 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 intermetallic magnets have applications for which prior art materials cannot even be considered.

Permanent magnet properties of bulk cobalt-rare earth intermetallic bodies are enhanced by reducing them to a powder. The as-ground powder can be incorporated in bonding media to produce a composite finished permanent magnet. Specifically, for most permanent magnet applications, cobalt-samarium intermetallic material is a powder having an average particle size ranging from about 1 micron or less to about 10 microns. As the particle size is increased, however, the permanent magnet properties of the material fall significantly. Specifically, direct reduction of a cobalt-samarium intermetallic bulk body to grains having a size as low as 50 microns, results in a material having such poor properties as to be useless for permanent magnet applications.

There are a number of disadvantages inherent in the use of a cobalt-samarium intermetallic powder having a particle size as low as 10 microns or lower. When this powder is exposed to air, particularly at temperatures slightly above room temperature, its intrinsic coercive force decreases irreversibly at a significant rate. This decay in coercive force substantially diminishes the advantages to be gained by converting the bulk cobalt-samarium intermetallic body to a powder. Also, preparation of powders of such fine size presents a number of handling problems and is time-consuming and costly.

The present invention overcomes the aforementioned disadvantages by providing novel large grain cobalt-samarium intermetallic permanent magnet material having permanent magnet applications. These permanent magnet properties are not shown by the as-ground grains of the same size. The time-consuming handling operations of transforming the material to a powder are eliminated. In comparison to the prior art powder, the grains of the present invention are easier to handle and they are more stable since they oxidize much more slowly. In addition, they show permanent magnet properties which are as good or better than the prior art powder. Because the present grains are significantly larger than the powder of the prior art, they are also easier to orient magnetically since magnetic alignment depends on a torque situation.

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 a graphical representation illustrating the permanent magnet properties of the material of the present invention and also showing the properties of the as-ground material.

FIG. 2 is a graphical representation illustrating the properties of the annealed grains of the present invention ranging in size from about 74 microns to 104 microns.

FIG. 3 is a graphical representation illustrating the properties of annealed grains ranging in size from 104 microns to 147 microns.

FIG. 4 is a graphical representation illustrating the properties of grains of the present invention of four different cobalt-samarium alloy compositions.

Briefly stated, the present process comprises casting an alloy of cobalt and samarium to produce a cast body having grains ranging in size from about 100 microns to 1000 microns. The cast body is annealed in an atmosphere in which it is substantially inert at a temperature ranging from about 900°C up to a temperature below its melting point for a period of time ranging from about 5 minutes to 24 hours. Generally, the annealing temperature ranges from about 900°C to 1200°C since no significant improvement in magnetic properties is produced at temperatures significantly higher than 1200°C. Annealing time for a particular annealing temperature depends on the particular permanent magnet properties desired. Specifically, to get significantly useful permanent magnet properties, it should be annealed at a particular annealing temperature for a period of time sufficient for the resulting free grains to show at room temperature, after being magnetized to at least approach saturation magnetization, i.e., within about 10 percent of full saturation magnetization, a relative magnetization value $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds with relative magnetization $4\pi J/B_r$, by definition, being 1.00 at zero demagnetizing field. The annealed body is then comminuted to a grain size corresponding to or smaller than the grain size of the cast body and ranging from about 50 to 200 microns. Alternatively, prior to annealing, the cast body can be comminuted to a grain size corresponding to its grain size or smaller than its grain size and the resulting free grains, ranging in size from about 50 to 200 microns, are annealed at a temperature ranging from about 900°C to 1200°C for a period of time ranging from about 5 minutes to 24 hours. Again, annealing time for a particular annealing temperature depends on the particular permanent magnet properties desired, and for significantly useful permanent magnet properties, the free grains should be annealed for a period of time sufficient for the annealed free grains to show at room temperature, after being magnetized to at least approach saturation magnetization, i.e., within about 10 percent of full saturation magnetization, a relative magnetization value of $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds with relative magnetization $4\pi J/B_r$, by definition, being 1.00 at zero demagnetizing field.

The cobalt-samarium alloy of the present invention contains samarium in an amount of about 34 to 38 percent by weight of the alloy, and generally, to attain the best magnetic properties, it contains samarium in an amount of about 35 percent by weight of the alloy. Grains produced in accordance with the present process but having a cobalt-samarium composition outside this range do not produce satisfactory permanent magnets. The alloy is prepared in an atmosphere in which cobalt and samarium are substantially inert such as a noble gas or under a vacuum by a number of methods such as, for example, by induction or arc melting the cobalt and samarium. The molten alloy should, preferably, also be cooled in an atmosphere in which it is substantially inert such as a noble gas or under a vacuum.

The cobalt-samarium alloy is cooled at a rate sufficiently slow to produce a solid cast body wherein the grains range in size from 100 to 1000 microns. This can be determined empirically using standard metallurgical techniques such as, for example, casting a liquid melt in a heated mold or simply allowing the molten alloy to cool in a crucible at room temperature. To prevent oxidation, cooling should be carried out in an atmosphere in which the alloy is substantially inert such as a noble gas or a vacuum. The solid cast body should have grains with a minimum size of about 100 microns since it would be difficult and not practical to try to obtain the required amount of the present single crystal free grains from a cast body wherein the grains are smaller than 100 microns.

The large grain solid cobalt-samarium intermetallic cast body can then be annealed or, alternatively, the cast body can be comminuted and the resulting free grains annealed. Annealing is carried out in an atmosphere in which the cobalt-samarium intermetallic material is substantially inert such as argon or in a vacuum. If the body is comminuted to free grains prior to annealing, the free grains also should be annealed in a container made of a material to which it is substantially inert such as molybdenum, tantalum or niobium to prevent contamination. Annealing can be carried out at a temperature ranging from about 900°C up to a temperature below the melting point of the material, and preferably, up to a temperature of 1200°C since no significant improvement in magnetic properties is produced at temperatures significantly higher than 1200°C. Generally, the best magnetic properties are produced at annealing temperatures ranging from 1100°C to 1200°C. The particular annealing time period for a particular annealing temperature depends largely on the specific permanent magnet properties desired. To produce significantly useful permanent magnet properties, it should be sufficiently long to produce an annealed material having the inherent property of showing at room temperature, after being magnetized to at least approach saturation magnetization, a relative magnetization value $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds. Generally, the longer the material is annealed, the higher its relative magnetization value becomes at higher demagnetizing fields, i.e. demagnetizing fields of −4 kilooersteds and higher. For example, in the present process, annealing the cobalt-samarium alloy at a temperature ranging from about 1100°C to 1200°C for a period of 10 hours should produce free grains having a relative magnetization value of at least 50 percent or 0.5 at a demagnetizing field of −10 kilooersteds. Generally, after an annealing period of 24 hours, no significant improvement in permanent properties occurs.

The term relative magnetization as used herein is the ratio of magnetization $4\pi J$ to remanent induction B_r. Specifically, when a magnetic field is applied to a permanent magnet material, a magnetization value of $4\pi J$ gauss is established therein. When the magnetic field is removed, the material has a remanent induction B_r. The intrinsic coercive force H_{ci} is the field strength at which the magnetization $4\pi J$ is zero and is a measure of a permanent magnet's resistance to demagnetization. An additional measure of a permanent magnet's resistance to demagnetization, and one which is useful in defining the permanent magnet properties of the present free grains, is the shape of the hysteresis loop in the second quadrant wherein magnetization $4\pi J$ or relative magnetization $4\pi J/B_r$ verses a negative field H which shows what positive values of magnetization can be maintained in the presence of the demagnetizing field H. Specifically, the more square this second quadrant curve is, the higher is the magnetization or relative magnetization at a particular negative or demagnetizing field H, and the greater is the resistance of the magnet to demagnetization at such demagnetizing field H.

It has been determined that the present cobalt-samarium alloy in solid bulk form has a saturation magnetization $4\pi J_s$ value of about 9000 to 11,000 gauss. This is the maximum magnetization value achievable for this cobalt-samarium composition in solid bulk form. Theoretically, in the ideal situation, free grains of this cobalt-samarium alloy, when incorporated in a non-magnetic matrix to a volume fraction of abut one-half, having an alignment factor of 1.00 and magnetized to saturation, should have a saturation magnetization $4\pi J_s$ of about 4500 gauss to 5500 gauss, a remanent induction B_r of about 4500 gauss to 5500 gauss and maintain a magnetization value of about 4500 gauss to 5500 gauss at a demagnetizing field of about −4 kilo-oersteds. In the present invention, when the free grains are incorporated in the non-magnetic matrix to a volume fraction of about one-half and magnetically aligned therein along their easy axis of magnetization so as to have an alignment factor of about 0.95 and magnetized to saturation, magnetization or approaching saturation magnetization, i.e. within about 10 percent of full saturation magnetization, the resulting permanent magnet has typically, a magnetization value $4\pi J$ of about 4000 gauss at a demagnetizing field of −4 kilooersteds. On the other hand, for significantly useful permanent magnet properties, the present free grains incorporated in a non-magnetic matrix to a volume fraction of one-half, i.e., comprising one-half by volume of the permanent magnet, and magnetized to saturation, or approaching saturation, should have, typically, a minimum magnetization value $4\pi J$ of about 2000 gauss at a demagnetizing field of −4 kilooersteds.

The rate at which the annealed material is cooled is not critical and a number of conventional techniques can be used which do not oxidize the material to any significant extent. Preferably, the annealed material is cooled in an atmosphere in which it is substantially inert such as, for example, argon or nitrogen, or it may be cooled in a vacuum, and generally, it is cooled to room temperature.

The cast body can be comminuted to free grains by a number of conventional methods such as, for example, by crushing by mortar and pestle, double disc pulverizer, or jaw crushers. Comminution is preferably carried out in an atmosphere in which the material is substantially inert such as argon or under a vacuum.

In the present invention, the grains of the cast cobalt-samarium body are single crystals and the cast body is comminuted to free grains having a size corresponding to the grain size of the cast body, or it is comminuted to free grains having a size smaller than the size of the cast body. Specifically, the free grains have a size ranging from about 50 microns to 200 microns. Free grains having a size significantly larger than 200 microns do not have useful permanent magnet properties. In addition, comminution should be carried out so that a major portion, i.e. at least 85 percent by weight of the free grains, are single crystal free grains. The single crystal structure of the free grains is determinable by standard metallographic techniques, such as, for example, X-ray diffraction techniques. Preferably, at least 95 percent by weight or substantially all of the resulting free grains are single crystal grains. Since the weakest bonds in the cast body exist at the grain boundaries, it is at these boundaries that breakage of the cast body usually preferentially occurs during comminution. In practice, due to breakage, the cast body should preferably have a grain size larger than that desired for the free grains to produce the highest amount of free grains of a single crystal.

The free grains of the present invention are incorporated in a non-magnetic matrix to form permanent magnets. To produce satisfactory magnetic alignment of the grains, the grains are incorporated into the non-magnetic matrix and, while the matrix is kept in a condition sufficiently liquid to maintain the grains in a substantially unlocked position, an aligning magnetizing field is applied to the incorporated grains to align then substantially along their preferred axis of magnetization which is the "C" or easy axis of magnetization, and, if desired, also to magnetize them as required. Specifically, since the grains are substantially unlocked in position, the incorporated single crystal grains subjected to the magnetizing field will be able to turn in a direction most favorable from a magnetic point of view, i.e., align along their easy axis of magnetization. While the magnetically aligned grains are still subject to the aligning magnetizing field, which should be at least 4 kilooersteds to produce satisfactory alignment, i.e., an alignment factor of at least about 0.85, the non-magnetic matrix is solidified to bond the grains and lock them in their magnetically aligned position. As used herein, the alignment factor is the ratio of the remanent induction B_r to the saturation magnetization $4\pi J_s$ multiplied by the volume packing fraction p. That is, $A = B_r/4\pi J_s \cdot p$. Frequently, in practice, an additional magnetizing field may be applied to the locked aligned grains to magnetize them to full saturation or to approach saturation magnetization and the specific strength of this magnetizing field depends largely on the degree of alignment of the grains. Generally, where the present grains have an alignment factor of at least about 0.85, such a field ranges from about 10 kilooersteds to 100 kilooersteds.

In another technique, if desired, the free grains of the present invention can be magnetized, to approach saturation, then incorporated in the liquid non-magnetic matrix, and an aligning magnetic field applied to the incorporated magnetized grains to align them along their easy axis of magnetization before the matrix is solidified to lock them in position.

The non-magnetic matrix used in forming the permanent magnets of the present invention can vary widely. It can be, for example, a plastic or resin, an elastomer or rubber, or a non-magnetic metal such as, for example, lead, tin, zinc, copper or aluminum. The extent to which the grains are packed in the matrix, i.e., the volume packing fraction of the present grains, can vary widely and depends upon the particular permanent magnet properties desired. Generally, the present free grains can be packed up to a maximum fraction of about 50 percent by volume.

Permanent magnets having useful permanent magnet properties for a wide range of applications are produced when the grains of the present invention are incorporated in a non-magnetic matrix and magnetized. Specifically, the resulting permanent magnets have a useful substantially stable magnetization $4\pi J$ in air at room temperature and generally at temperatures ranging from room temperture up to about 75°C. The permanent magnets of the present invention are useful in telephones, electric clocks, radios, television, and phonographs. They are also useful in portable appliances, such as electric toothbrushes and electric knives, 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.

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 grain structure of the solid cast cobalt-samarium body was determined by slicing off a portion of the casting, polishing it and examining it under a microscope.

All annealing was carried out in an inert atmosphere of purified argon and upon completion of the annealing, the annealed product was transferred to a chamber having an atmosphere of argon at room temperature where it cooled to room temperature.

The solid cast cobalt-samarium body was comminuted in an atmosphere of nitrogen at room temperature by means of double disc pulverizer.

The size of the free grains was determined by standard techniques using U.S. Standard Screen Sieves.

Samples referred to as as-ground samples were not annealed.

All magnetic measurements were carried out at room temperature.

To determine the magnetic properties of each sample of grains, the grains were incorporated into a body of molten liquid paraffin wax in a small glass tube to a fraction of about 50 percent by volume. The wax was sufficiently liquid so that the grains were substantially unlocked in position. An aligning magnetic field was then applied to the incorporated sample to align the grains along their easy axis of magnetization and the wax was cooled in the aligning magnetic field until it solidified to lock the magnetically aligned grains in position.

Under the conditions set forth in the following examples, the resulting alignment factor was at least about 0.85 and the grains were magnetized to at least approach saturation magnetization, i.e. within about 10 percent of full saturation magnetization.

Relative magnetization $4\pi J/B_r$ was measured at demagnetizing fields starting from zero demagnetizing field. At zero demagnetizing field, relative magnetization $4\pi J/B_r$ has, by definition, a value of 1.00.

EXAMPLE 1

About 500 grams of an alloy melt of 63 percent cobalt and 37 percent samarium was prepared by induction-melting under purified argon in an alumina crucible which had an inner diameter of about 2 inches and was about 3 ½ inches high. The liquid melt filled about one-half of the crucible and was kept in an argon atmosphere at room temperature to slowly solidify. To recover the resulting solid cast alloy, the crucible was broken with a hammer. The grains in the cast alloy ranged in size from abuot 100 microns to 1000 microns.

The cast alloy was comminuted in a nitrogen atmosphere by means of a double disc pulverizer to produce a batch of free grains having a size ranging from about 74 microns to 104 microns with about 95 percent of these grains being single crystal free grains. A portion of this batch was separated and used to form Sample A to determine the properties of the as-ground material. The remaining portion of this batch of free grains was spread out in a tantalum tray and annealed at a temperature of 1120°C for 15 minutes. A portion of the annealed grains was used to form Sample B.

An aligning magnetizing field of 12 kilooersteds was applied to each sample. The resistance to demagnetization of each sample at particular demagnetizing fields was then measured at room temperature. The results are shown in FIG. **1**. Specifically, FIG. **1** shows what positive values of magnetization can be maintained in the presence of the demagnetizing field H. From FIG. **1** it can be seen that Sample A, the as-ground sample, has poor permanent magnet properties and is completely demagnetized at a demagnetizing field of −2.8 kilooersteds which makes it useless for most permanent magnet applications. In contrast, Sample B which illustrates the present invention, is not demagnetized at a demagnetizing field in excess of −10 kilooersteds, and at a field of −4 kilooersteds, it has a relative magnetization value higher than 50 percent which makes it useful for a wide range of permanent magnet applications such as, for example, as a moving magnet meter.

EXAMPLE 2

The procedure used in this example was the same as that set forth in Example 1 except for a difference in annealing time, annealing temperature and aligning magnetizing field. Specifically, Sample C was formed from as-ground grains. The grains of Sample D were annealed at 1100°C for one-half hour. The grains of Sample E were annealed at 1100°C for one-half hour, furnace cooled to 900°C and annealed at 900°C for **1** hour. The grains of Sample F were annealed at 1100°C for one-half hour, furnace cooled to 900°C and annealed at 900°C for 14 hours.

An aligning magnetizing field of 17 kilooersteds was applied to each sample. The resistance to demagnetization of each sample was then measured at room temperature at particular demagnetizing fields. The results are shown in FIG. **2**. From FIG. **2** it can be seen that the as-ground Sample C had poor permanent magnet properties and was completely demagnetized at a demagnetizing field of about −2.8 kilooersteds. In contrast, Samples D, E and F, which illustrate the present invention, have permanent magnet properties which make them useful for a wide range of permanent magnet applications as shown by the high magnetization values that these samples can maintain at demagnetizing fields of −4 kilooersteds and higher.

EXAMPLE 3

The procedure used in this example was the same as that set forth in Example 1 except for a difference in free grain size, annealing time, annealing temperature and aligning magnetizing field. Specifically, the batch of free grains in this example ranged in size from 104 microns to 147 microns with about 95 percent of these grains being single crystal free grains. Sample G was formed from as-ground grains. The grains of Sample H were annealed at 1100°C for one-half hour. The grains of Sample I were annealed at 1100°C for one-half hour, furnace cooled to 900°C and annealed at 900°C for 1 hour. The grains of Sample J were annealed at 1100°C for one-half hour, furnace cooled to 900°C and annealed at 900°C for 14 hours. An aligning magnetizing field of 17 kilooersteds was applied to each sample. The resistance to demagnetization of each of the samples is shown in FIG. **3**. From FIG. **3** it can be seen that the as-ground Sample G had poor permanent magnet properties and was completely demagnetized at a demagnetizing field of about −1.3 kilooersteds. On the other hand, significantly useful permanent magnet properties were shown only by Sample J which showed a relative demagnetizing value of at least 50 percent at a demagnetizing field of −4 kilooersteds.

EXAMPLE 4

In this example four cobalt-samarium cast alloys were prepared in the same manner as set forth in Example 1. The alloy used to form Sample K was 63 percent cobalt-37 percent samarium, Sample L was 64 percent cobalt-36 percent samarium, Sample M was 65 percent cobalt-35 percent samarium and Sample N was 66 percent cobalt-34 percent samarium. The grains in each solid cast alloy ranged in size from 100 microns to 1000 microns.

Each cast alloy was comminuted at room temperature in a nitrogen atmosphere by means of a double disc pulverizer to produce free grains having a size ranging from 104 microns to 147 microns. A sample of free grains of each alloy batch was annealed at 1100°C for 16 hours. Each annealed sample of free grains was pre-magnetized at room temperature with a magnetizing field of 60 kilooersteds before being incorporated into the body of molten paraffin wax. An aligning magnetizing field of 17 kilooersteds was applied to each incorporated sample of grains. The resistance to demagnetization of each of the samples is shown in FIG. **4**. All of the samples plotted in FIG. **4** illustrate the present invention and show what high magnetization values can be maintained at high demagnetizing fields making them useful for a wide range of permanent magnet applications.

In copending U. S. Pat. application Ser. No. 244,423, entitled "Large Grain Cobalt-Samarium Permanent Magnet Material Stabilized With Zinc And Process" filed of even date herewith 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, the present grains are disclosed along with a zinc treatment which stabilizes or improves the permanent magnet properties of the grains of the present invention at elevated temperatures.

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

1. A process for preparing large single crystal free grains of cobalt-samarium alloy having useful permanent magnet properties which comprises forming an alloy melt of cobalt and samarium wherein the samarium content ranges from about 34 to 38 percent by weight of said alloy, cooling said alloy melt at a rate sufficiently slow to produce a solid cast body wherein the cast grains have a size ranging from about 100 microns to 1000 microns, said cast grains being single crystal grains, annealing said solid cast body in an atmosphere in which it is substantially inert at a temperature ranging from 900°C to 1200°C for a period of time ranging from about 5 minutes to 24 hours, and comminuting said annealed cast body to produce free grains having a size corresponding to or smaller than the size of said cast grains, recovering said free grains ranging in size from about 50 to 200 microns with at least 85 percent by weight of said recovered free grains being single crystal free grains, said annealing being carried out so that said resulting recovered free grains have the property of showing at room temperature after being magnetized to at least within about 10 percent of full saturation magnetization a relative magnetization value $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds.

2. A process according to claim **1** wherein said annealing temperature ranges from about 1100°C to 1200°C.

3. A process according to claim **1** wherein at least 95 percent by weight of said recovered free grains are single crystal grains.

4. A process for preparing a solid permanent magnet which comprises incorporating said recovered free grains of claim **1** in a non-magnetic matrix having a consistency which is sufficiently liquid to leave said grains in a substantially unfixed position, applying to said incorporated grains a magnetic field of at least 4 kilooersteds to align the grains along their easy axis of magnetization and fixing said magnetized grains in their magnetically aligned position by solidifying said non-magnetic matrix material.

5. A process according to claim **4** wherein said non-magnetic matrix material is selected from the group consisting of plastics, elastomers, metals and wax.

6. A process for preparing large single crystal free grains of cobalt-samarium alloy having useful permanent magnet properties which comprises forming an alloy melt of cobalt and samarium wherein the samarium content ranges from about 34 to 38 percent by weight of said alloy, cooling said alloy melt at a rate sufficiently slow to produce a solid cast body wherein the cast grains have a size ranging from about 100 microns to 1000 microns, said cast grains being single crystal grains, comminuting said solid cast body to produce free grains having a size corresponding to or smaller than the size of said cast grains, recovering said free grains ranging in size from about 50 to 200 microns with at least 85 percent by weight of said recovered free grains being single crystal free grains, and annealing said recovered free grains in an atmosphere in which they are substantially inert at a temperature ranging from 900°C to 1200°C for a period of time ranging from about 5 minutes to 24 hours, said annealing being carried out so that the annealed recovered free grains have the property of showing at room temperature after being magnetized to at least within about 10 percent of full saturation magnetization a relative magnetization value $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds.

7. A process according to claim **6** wherein at least 95 percent by weight of said recovered free grains are single crystal free grains.

8. A process for preparing a solid permanent magnet which comprises incorporating said annealed free grains of claim **6** in a non-magnetic matrix having a consistency which is sufficiently liquid to leave said grains in a substantially unfixed position, applying to said incorporated grains a magnetic field of at least 4 kilooersteds to align the grains along their easy axis of magnetization and fixing said magnetized grains in their magnetically aligned position by solidifying said non-magnetic matrix material.

9. A process according to claim **8** wherein said non-magnetic matrix material is selected from the group consisting of plastics, elastomers, metals and wax.

10. Annealed free grains consisting essentially of cobalt-samarium alloy wherein the samarium content ranges from about 34 to 38 percent by weight of said cobalt-samarium alloy, said annealed free grains ranging in size from about 50 microns to 200 microns with at least 85 percent by weight of said free grains being single crystal grains, said annealed free grains having the property of showing at room temperature after being magnetized to at least approach saturation magnetization a relative magnetization value $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds where $4\pi J$ is the magnetization value and B_r is the remanent induction.

11. Annealed free grains consisting essentially of cobalt-samarium alloy containing about 35 percent by weight samarium, said annealed free grains having an average size of about 100 microns to 150 microns with at least 95 percent by weight of said free grains being single crystal grains, said annealed free grains having the property of showing at room temperature after being magnetized to at least approach saturation magnetization a relative magnetization $4\pi J/B_r$ of at least 50 percent at a demagnetizing field of −4 kilooersteds where $4\pi J$ is the magnetization value and B_r is the remanent induction.

12. A permanent magnet having as an active magnetic component said annealed free grains of claim **10** and wherein said annealed free grains are incorporated in a non-magnetic matrix.

13. A permanent magnet having as an active magnetic component said annealed free grains of claim **11** and wherein said annealed free grains are incorporated in a non-magnetic matrix.

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