METHOD AND APPARATUS FOR PRODUCING FINE-PARTICLE PERMANENT MAGNETS

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ABSTRACT

Fine-particle permanent magnets are prepared by hydrostatically driving a pre-pressed member composed of powder of magnetically aligned particles through a die opening of diminishing cross-section, so as to cause compression of the pre-pressed member into a permanent magnet form wherein the hydrostatic force is supplied by a surrounding liquid medium under a compressive force.

15 Claims, 2 Drawing Figures
METHOD AND APPARATUS FOR PRODUCING FINE-PARTICLE PERMANENT MAGNETS

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates generally to a method and apparatus for producing fine-particle permanent magnets, and more particularly to a method and apparatus for producing fine-particle permanent magnets from an intermetallic compound of a 3d transition element and a rare earth metal, without sintering.

2. Description of the Prior Art

It is known to prepare fine-particle permanent magnets from intermetallic compounds of a 3d transition metal, such as the Group VIII metals including cobalt, iron or nickel, in combination with a rare earth metal of the Lanthanide series. By the term “3d transition element” is intended to mean a member of the first transition metal series. For instance, in U.S. Pat. 3,424,578, there is disclosed a fine-particle permanent magnet prepared from an intermetallic compound of the form RX, wherein R is a rare earth of the Lanthanide series, such as Y, La, Ce, Pr, Nd or Sm, or mixtures thereof, and A is Co, Fe, Mn of mixtures thereof. Several different rare earth metals, for instance, may be incorporated into the same crystall lattice. Permanent magnets prepared from these types of ferro or ferrimagnetic compounds are known to possess extremely high remanence, high magnetic crystal anisotropy and very high coercive force, and hence, are considered to be quite desirable for many specialized purposes.

In the usual method for preparing fine-particle permanent magnets, an intermetallic compound is first produced by conventional foundry metallurgical techniques. This compound is then ground into a powder having a grain size of about several microns. The powder is aligned in a magnetic field and pressed into a unitary structure. This structure is then sintered at a temperature of from about 1,050° to 1,100° C. for a short period of time, in an inert atmosphere.

One difficulty with this prior art technique, however, is the finding that sintering tends to deteriorate the magnetic properties of the intermetallic compound. It is now believed that this deterioration is caused by the presence of a relatively large quantity of oxygen which has been adsorbed into the powder during the earlier processing procedures. Even when sintering is conducted in an inert atmosphere, therefore, the adsorbed oxygen is frequently present in sufficient quantities to effect reaction with the rare earth metal in the intermetallic compound and thereby cause disintegration of the crystal structure of the powder and inactivation of the rare earth metal.

Another undesirable aspect of the prior art technique is that sintering is economically and technically an undesirable procedure, which has severely hindered the successful development of a continuous method for producing such permanent magnets.

Accordingly, a need exists for a method whereby fine-particle permanent magnets may be produced without the undesirable effects caused by conventional sintering steps.

SUMMARY OF THE INVENTION

Accordingly, it is one object of this invention to form fine-particle permanent magnets whereby crystal structure disintegration of the powder and inactivation of the rare earth metal may be avoided.

It is another object of this invention to provide a method and apparatus for producing fine-particle permanent magnets from intermetallic compounds without deteriorating the magnetic properties of the intermetallic compound powder.

It is still a further object of this invention to provide fine-particle permanent magnets whereby the sintering procedure, required of prior art methods, can be avoided.

These and other objects, which will hereinafter become more apparent, have now been attained by hydrostatically driving a pre-pressed member composed of a powder of magnetically aligned particles through a die opening of diminishing cross-section, so as to cause compression of the pre-pressed member into a permanent magnet form, wherein the hydrostatic force is supplied by a surrounding liquid medium under a compressive force.

BRIEF DESCRIPTION OF THE DRAWINGS

Various other objects, features and attendant advantages of this invention will be more fully appreciated as the same becomes better understood from the following detailed description when considered in connection with the accompanying Drawings, in which:

FIG. 1 is a diagrammatic cross-section of a device especially adapted for carrying out the method of this invention which is characterized by a large hydrostatic pressure-applying means for surrounding and driving a powder member through a die opening of diminishing cross-section, and another hydrostatic means for surrounding and applying a counteracting pressure of lesser proportion to the further compacted powder form exiting from the die; and

FIG. 2 is a diagrammatic cross-section view of a container die positioned in a magnetic field and illustrates a magnetic powder being drawn therethrough for providing the compacted member of aligned magnetic particles shown in FIG. 1.

DETAILED DESCRIPTION OF THE INVENTION

According to the invention, an intermetallic compound powder member is pre-pressed and the particles thereof are magnetically aligned and thereafter the pre-pressed, magnetically aligned powder member is cold worked by driving the same through a die opening of diminishing cross-section by a liquid, under pressure, acting on all sides of the powder member as the propelling force for driving the member through the die. This technique is somewhat analogous to hydrostatic extrusion, which has been used in the prior art for compacting various other types of materials. Heretofore, conventional extrusion techniques, however, have never been considered for powders of intermetallic compounds, largely because of the known high brittleness of such compounds. To this effect, one of the important attributes of the present invention is the realization that when a highly pressurized liquid is caused to surround the compacted powder and is utilized as the propelling force for driving a powder member through a die, it acts with the deformation forces of the die to enable
the brittle powder member to become ductile and de-
formable.

Any liquid which is inert in the system may be used
herein, such as, for instance, any of a variety of mineral
or petroleum oils.

The propelling pressure may advantageously be
maintained in the system at between about 12 to 20
kbar.

It is also desirable to provide a counteracting liquid
pressure at the small diameter cross-section, or outlet
of the die, which will completely surround and exert a
lesser pressure on the compacted powder emerging
from the die, in order to reduce still further the risk of
cracking of the brittle powder during deformation.

Good results are obtained when the counteracting
liquid is under a pressure of approximately one-half of
the value of that of the first liquid.

It is desirable to use a conical die having an angle of
inclination of less than, or equal to, approximately 60°,
so as to prevent the build-up of excessive deformation
forces there-in.

In preparing the pre-pressed powder member used in
this invention, an intermetallic compound, as described
above, is produced by ordinary foundry metallurgy
techniques. This compound is ground into a powder of
several microns in size and is introduced into a highly
ductile container. The container is completely filled
with the powder and the entire container with its pow-
der content is drawn through a first conical die. This
expedient tends to lower the overall production costs
by protecting the magnetic material from corrosive and
mechanical attack, and by forming a very smooth solid
surface which requires no subsequent machining.

Prior to entrance through the die, or at the deforming
portion of the die, the powder within the container is
subjected to a magnetic field of sufficient magnitude to
cause alignment of the powder. The aligning field may
extend parallel to the direction of the container move-
ment. Compaction of the magnetic powder in the die,
to a first degree, while simultaneously subjecting the
powder to magnetic alignment in the drawing direction,
will result in a well-developed homogeneous alignment
of the particles within the compacted powder.

The pre-pressed powder member is then subjected to
the hydrostatic extrusion process of this invention.

Using this technique, extremely high degrees of de-
formation are possible, even approaching 100 percent of
the theoretical density for the particular material being
treated. This degree of intensive deformation is quite
advantageous in that it provides a high degree of
homogeneity throughout the product permanent mag-
net, so that applied mechanical stresses are distributed
homogeneously throughout, and the very high resulting
magnetic values are distributed homogeneously
throughout.

The methods of this invention can be used to allow
continuous production of fine-particle permanent mag-
nets at a low cost, or can be used for quasi-continuous
production of diverse dimension bar magnets, or the
like, which can be provided to very close tolerances.

What is especially quite interesting is the fact that
when using the methods of this invention, there is mini-
mal destruction of the magnetic orientation of the pow-
der particles.

Referring now to the drawings, and more particularly
to FIG. 1, wherein is shown a die member 4 having a
continuous opening composed of a conical, tapered
inlet portion 2 of decreasing cross-section, and an out-
let portion 3, being substantially tubular in configura-
tion. A pre-pressed powder member 1 is shown being
driven through the die 4, whereby it is compressed to
about 100 percent of its theoretical density. The com-
 pacted powder member then emerges through the die
outlet section 3.

The die 4 is disposed in one end of a high-pressure
cylindrical vessel 9 which also contains a liquid me-
dium 5, such as an oil which surrounds the inlet por-
tion 2 of the die 4. The liquid 5 may be maintained
under a fairly high pressure of from about 12 to 20 kbar
by means of a plunger 11 movably disposed in the other
end of the high pressure vessel 9, being spaced at a dis-
tance in advance of the inlet portion 2 of the die 4, and
thereby is effective to propel the powder member 1
through the die. A pressure equalizing valve 10 may be
provided, if desired, in the liquid medium 5 in the cylin-
drical vessel 9 intermediate the plunger 11 and the die
member 4.

A second substantially cylindrical vessel 12 is se-
 cured to the high pressure vessel 9, having a portion
thereof disposed within the end of the high pressure
vessel 9 opposite the plunger 11 for closing the vessel
9 and supporting the die 4 therein with the outlet por-
tion 3 being open into the vessel 12. The vessel 12 is
filled with a liquid medium 6, which may also be an oil,
for receiving and completely enveloping the com-
 pacted powder member 1 exiting from the outlet 3 of
the die 4. Preferably, the liquid 6 in the vessel 12 is
maintained under a pressure substantially less than that
applied to the liquid medium 5 in the vessel 9, for ex-
ample, about one-half thereof, or approximately be-
 tween 6 and 10 kbars, by a plunger 13 movably dis-
posed in an opposite portion thereof, for providing a
counter-propelling force on the powder member 1.

In the illustrated embodiment, the angle of inclina-
tion, designated by the reference numeral 8, of the con-
cical inlet portion 2 of the die 4, preferably, is not more
than 60°.

Using the above-discussed technique, it has been pos-
sible to successfully produce fine-particle permanent
magnets of the intermetallic compound SmCo5.

Referring now to FIG. 2, first the powder member 1,
shown being formed into a permanent magnet in FIG.
1 above, may first be precompressed and have the par-
ticles thereof magnetically aligned by placing the pow-
der in a highly ductile container 14, and drawing the
same through a die 15 disposed in a magnetic field. In
one embodiment, the container 14 consists of a low
carbon content stainless steel, such as 304 S.S., 304 L
or 305 steel (AISI-SAE standard 18/8 steels of very low
carbon content). The thickness of the container wall in
this instance was 0.1 mm.

The entire container with the powder contents is
drawn through the die, or drawing block 15. This die
was suitably prepared from M3 or M4 grade tool steel
(AISI-SAE standard). In this example, the container 14
is held and pulled through the die by a clamp 19.

To initiate the drawing operation, the container 14 is
filled with the powder and then pulled at one end to
form a point or tip, which is passed through the die
block 15 to be engaged by clamp 19 and the remainder
of the container is drawn through the die block 15 by
moving clamp 19 away from block 15 as indicated by
the arrow in FIG. 2.
In this example, the die entrance had a cross-sectional diameter of 10 mm and the die outlet had a cross-sectional diameter of 6-7 mm. The container, of course, had the same outer diameter after being drawn through the die block 15, as the outlet diameter of the die.

If desired, the container can be drawn repeatedly through a series of blocks of decreasing cross-sections to achieve very high degrees of compaction. It is preferable to use a slow drawing speed, and particularly between 3 to 30 meters per minute. For economical considerations, the draw length should be at least 1 meter, since the beginning portions and end portions are generally unusable.

The drawing angle 16 of the conical portion of the die 15 should not exceed about 7-9'. Of course, the particular degree of the angle will depend upon the desired degree of deformation. The smaller the angle, the smaller will be the degree of reduction.

The die block 15 in FIG. 2 is shown as being surrounded by a magnetic coil 17 to which a current may be applied for inducing a magnetic flux which produces a magnetic field parallel to the direction of the draw and the movement of the container 14. The field extends from a point prior to the entrance of the container 14 into the die block 15 such that the particles of the powder are still in a relatively loosely packed state, and extends across the die block adjacent that portion of the die at which the reduction is discontinued. A magnetic shield 18 surrounds the coil 17 to define the extent of the container 14 exposed to the magnetic field. It has been found that when the powder particles are magnetically aligned during this period, they may be compacted by being drawn through the die block 15 while strictly maintaining their magnetic orientation.

Having now fully described the invention, it will be apparent to one or ordinary skill in the art, that many changes and modifications can be made thereto without departing from the spirit and scope of the invention.

What is claimed and desired to be secured by letters patent of the United States is:

1. A method of producing fine-particle permanent magnets from intermetallic compounds of a 3d transitional element and a rare earth element, comprising the steps of:
   - precompressing and magnetically aligning the particles of a powder of said intermetallic compound;
   - enveloping said pre-compressed and magnetically aligned particles at the inlet of a die of diminishing cross-section with an inert liquid, and
   - driving said aligned powder through said die of diminishing cross-section by means of said inert liquid so as to further compress said powder into a compacted form.

2. The method as set forth in claim 1 further comprising the step of maintaining said liquid at said inlet under a pressure of from about 10 to 20 kbar.

3. The method as set forth in claim 1 further comprising the step of providing a counter-acting pressure on the emerging compacted form at the outlet of said die by means of a surrounding inert liquid maintained under pressure.

4. The method as set forth in claim 3 further comprising the step of maintaining said inert liquid at said outlet of said die at a pressure of approximately one half of said pressure of said liquid at said inlet.

5. The method as set forth in claim 4 further comprising the step of reducing the diameter of said powder by substantially fifty percent as said powder is driven through said die.

6. A method of producing fine-particle permanent magnets from intermetallic compounds of a 3d transitional element and a rare earth element, which comprises the steps of:
   - encapsulating a powder of said intermetallic compound in a ductile container;
   - precompressing and magnetically aligning the particles of said powder within said container;
   - enveloping said container at the inlet of a die of diminishing cross-section with an inert liquid, and
   - driving said container through said die of diminishing cross-section by means of said inert liquid so as to further compress said powder into a compacted form.

7. The method as set forth in claim 6 wherein said step of pre-compression is accomplished by drawing said ductile container through a die at the rate of about 3 to 30 meters per minute, and wherein said draw length is at least one meter.

8. An apparatus for producing fine-particle permanent magnets from intermetallic compounds of a 3d transitional element and a rare earth element, comprising:
   - means for precompressing and magnetically aligning the particles of a powder of said intermetallic compound;
   - a die of diminishing cross-section having an inlet portion and an outlet portion;
   - means for surrounding said inlet portion of said die with an inert liquid; and
   - means for applying pressure to said liquid for driving said magnetically aligned powder through said die to further compress said powder into a compacted form.

9. An apparatus as set forth in claim 8, further comprising:
   - means exposed to said outlet portion of said die for providing liquid in surrounding relation with said compacted powder form exiting from said outlet portion; and
   - means for applying pressure to said means exposed to said outlet portion of said die.

10. An apparatus as set forth in claim 8, wherein the angle of inclination of said inlet portion of said die is not more than 60°.

11. An apparatus as set forth in claim 8, wherein said means for pre-compressing said powder comprises:
   - a second die having an inlet portion of diminishing cross-section and an outlet portion;
   - means for drawing said powder through said die; and
   - means for establishing a magnetic field about at least said inlet portion of said die for aligning the particles of said powder in the direction through which said powder is drawn.

12. An apparatus as set forth in claim 11, wherein said magnetic field extends substantially from a position immediately prior to said inlet portion of said second die through said inlet portion.

13. An apparatus as set forth in claim 12, further comprising means for shielding said means for establishing a magnetic field.

14. An apparatus as set forth in claim 11, wherein the drawing angle of said inlet portion of said second die is less than approximately 9°.

15. An apparatus as set forth in claim 8 further comprising a very low carbon content stainless steel container for encapsulating said powder.