June 27, 1933. E. E. SCHUMACHER ET AL 1,915,386

PROCESS FOR THE PRODUCTION OF MAGNETIC MATERIALS IN POWDERED FORM

Filed April 16, 1932

FIG. 1

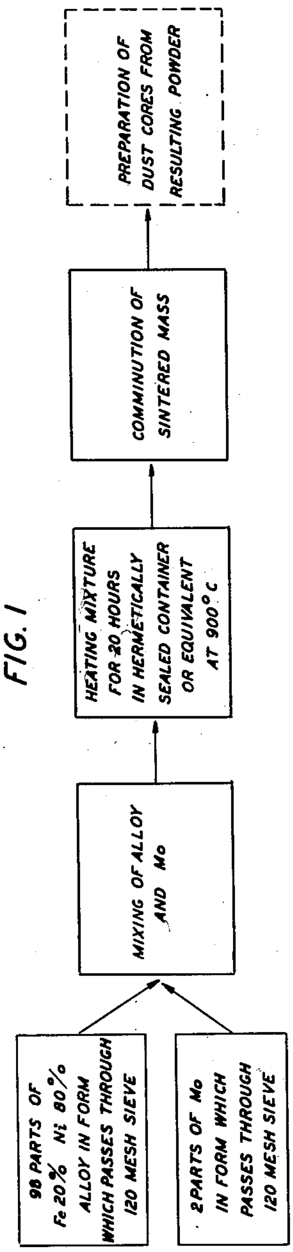
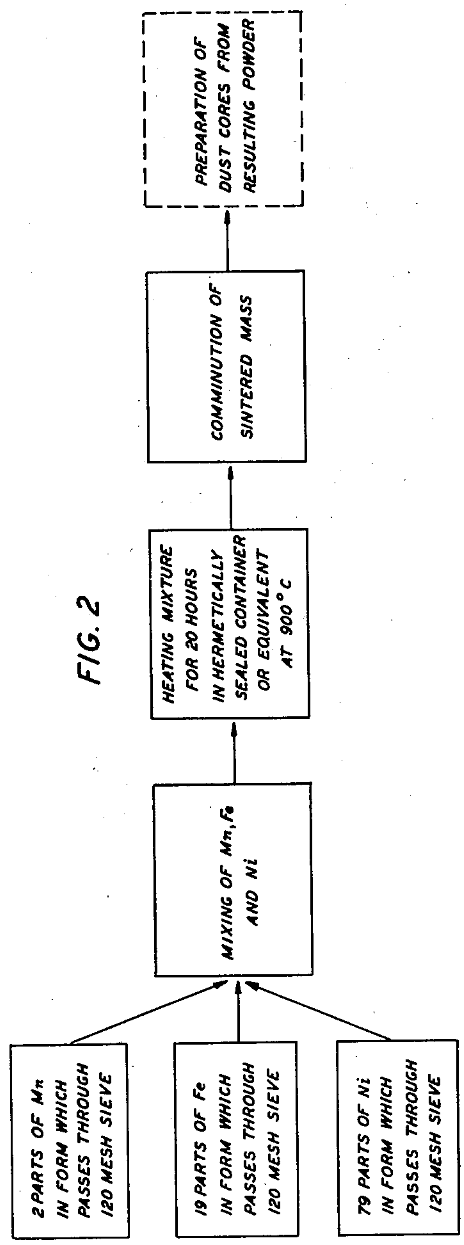


FIG. 2



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UNITED STATES PATENT OFFICE

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PROCESS FOR THE PRODUCTION OF MAGNETIC MATERIALS IN POWDERED FORM

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The present invention relates to the production of magnetic materials and more particularly to methods of producing such materials comprising a plurality of elements in finely divided form.

An object of the invention is to facilitate the production of finely divided magnetic materials which are employed as cores for loading coils and other induction devices.

Another object of this invention is to enable production of magnetic materials in finely divided form from the elemental constituents of these materials.

A more particular object of this invention is to provide a more desirable method than has heretofore been available for the preparation in finely divided form of magnetic alloys comprising iron and nickel, iron and cobalt, or iron, cobalt and nickel together with a third constituent such as manganese, a metal of the chromium-molybdenum-tungsten group or silicon.

The improvement of the electrical and magnetic characteristics of iron-nickel, iron-nickel-cobalt and iron-cobalt alloys by the addition of certain other constituents is well known in the art. The addition of small quantities of chromium, molybdenum, tungsten, manganese or silicon has been particularly advantageous in this respect. However, the reduction to powdered form of alloys required for dust cores of magnetic compositions containing these elements has been found to be fraught with difficulty. The physical properties of iron-nickel, iron-nickel-cobalt and iron-cobalt alloys containing these elements are especially critical to the presence of impurities, such as sulphur and oxygen usually present in ferrous alloys. Sometimes these alloys due to the quantity of impurities become "hot-short" and consequently too brittle for the hot rolling operation in the method now in use which is hereinafter described. On the other hand, if these impurities are not present in a quantity which produces the "hot-short" effect, the alloys are too tough for pulverizing.

In the method now in use for the production of a dust core, the constituent elements which comprise the alloy for the dust core are first melted together to form a homogeneous alloy. The alloy is then cast and the resulting ingot hot-rolled to the desired dimensions. As stated previously, if the alloy is hot-short, serious difficultv is encountered in this operation. After the alloy has been rolled, it is placed in a ball mill or other means for reducing the strips to powdered form. In this operation if the alloy is too tough, it cannot be readily pulverized. Inasmuch as strains are produced in the alloy by the pulverizing, the powder is annealed. The powder is then insulated with a suitable material and subsequently pressed into cores. The cores are again annealed and are ready for use for coils.

According to one feature of this invention, an elemental constituent is alloyed with the binary and ternary alloys of iron, nickel and cobalt to produce a magnetic material in powdered form. The constituent and the alloy are pulverized to the desired degree of fineness. The two are mixed together and placed in a container which is subsequently hermetically sealed. The container is heated to a temperature below the fusion points of the alloy and the element but sufficiently high to result in the substantial diffusion of the element and the alloy. The resulting sintered mass is readily broken up into the desired pulverized form. This method obviates the difficulties encountered as a result of the physical characteristics of the ferrous alloys containing manganese, silicon or a metal of the chromium-molybdenum-tungsten group. The binary and ternary alloys of iron, nickel and cobalt may be prepared by the fusion method and after the pulverization mixed with the powder of a constituent or constituents of the chromium-molybdenum-tungsten group, manganese or silicon and heated in hermetically sealed containers at a temperature that produces substantial diffusion. The sintered mass may then be broken up, and made into cores as in the method now employed. Ternary alloys of iron, nickel and cobalt may be produced in powdered form by this method by mixing in powdered form two binary alloys or one binary alloy and the other element of the alloy or the three constituents of the alloy and treating the mixture in a manner similar to that described. For example, to prepare an alloy comprising iron, nickel and cobalt in powdered form, a mixture of an alloy comprising nickel and iron and another comprising iron and cobalt, both of which are in powdered form or a mixture of powdered alloy comprising iron and cobalt and powdered nickel or a mixture of powdered iron, nickel and cobalt in their elemental state may be employed.

According to another feature of this invention, the elemental constituents of the alloys desired may be mixed together in powdered form and heated to a temperature below the fusion points of the lowest melting constituent and high enough for the constituents to diffuse into each other. The sintered mass may then be broken up to the desired degree of fineness. In this manner iron-nickel-chromium, iron-nickel-molybdenum, iron-cobalt-nickel-tungsten alloys or any other binary, ternary or quaternary magnetic alloy may be produced in powdered form.

The nature of this invention is indicated by the accompanying drawing in which Fig. 1 represents in schematic form the steps involved in the preparation of magnetic alloys for dust cores from an element and a metal comprising one or more constituents and in which Fig. 2 shows schematically the process for the production of a ternary alloy in powdered form from three elemental constituents. In Figs. 1 and 2, the steps in the preparation of a comminuted alloy are indicated in boxes of solid black lines, while the usual subsequent operations for producing the cores from the powdered dust are shown in the boxes of dotted lines.

Two specific examples serve to demonstrate the procedure of this method. It is understood that this invention is not restricted to the particular proportions stated, or to the details of manipulations described, or to apparatus employed, or to the temperatures attained, or to the period of time of heating, or to the degree of fineness of the alloys or constituents used, all of these factors being merely given by way of illustration.

The first example involving the production of a comminuted alloy comprising iron, nickel and molybdenum illustrates the method for the preparation of a finely divided alloy from a powdered metal comprising one or more elements and a powdered element. The method of producing the finely divided iron-nickel-molybdenum alloy is shown schematically in Fig. 1. An alloy comprising approximately 80% nickel and the remainder chiefly iron is pulverized in an attrition mill or other mechanical comminution agency to a powder fine enough to pass through a 120-mesh sieve. Powdered molybdenum obtained by either pulverizing or chemical methods in a form finer than 120-mesh is mixed with the iron-nickel alloy in the proportions of 2 parts of molybdenum and 98 parts of iron-nickel alloy. The mixture of the iron-nickel alloy and the molybdenum is placed in a container provided with a cover. At times, it is desirable to place a small layer of iron filings on top of the mixture. This practice is particularly advantageous if the mixture occupies only a portion of the container. After the mixture is within the container, the cover is placed thereon. The junction between the cover and the container is sealed with a refractory cement or other sealing material. This latter procedure affects the substantial hermetically sealing of the container for metallurgical purposes. It is to be understood, however, that the mixture may be placed in any instrumentality that will insure the substantial diffusion of the molybdenum and the iron-nickel alloy and prevent material contamination of the resulting molybdenum-nickel-iron alloy. The mixture in the container is heated in the range between 800° C. to 1200° C. for a period of approximately twenty hours, the desirable temperature for this particular alloy being about 900° C. The resulting product is a sintered mass which can be readily broken up into the desired powdered form by any well known comminution method. In the preparation of the alloy of this example, an attrition mill is employed. The powder is then insulated, pressed and annealed in the same manner as cores prepared by the fusion method. Cores containing iron-nickel-molybdenum alloys produced by this method have substantially the same magnetic and electrical characteristics as those prepared from approximately the same amount of constituents by the fusion method. Cores containing iron-nickel-chromium, iron-nickel-tungsten, iron-nickel-manganese and iron-nickel-silicon alloys in powder form as well as iron-cobalt and iron-nickel-cobalt alloys containing a third element are examples of magnetic alloys which can be produced by this method.

The second example demonstrates the method for the preparation of an alloy in finely divided form from its elemental constituents. In the preparation of alloys from their elemental constituents, the method is substantially the same. For example, if an alloy of approximately the same composition as that prepared in the first example is desired, except that managanese is substituted for molybdenum, the following procedure indicated schematically in Fig. 2 is adopted: Approximately 2 parts of manganese are mixed with 79 parts of nickel and 19 parts of iron, all three of which pass through a 120-mesh sieve. These powdered metals may be obtained in a finely divided state by pulverizing the rolled sheet, or casting as described above, or by chemical processes, such as electrolysis or in the case of nickel and iron from their respective carbonyls. The mixture is placed in a container which is subsequently hermetically sealed according to the procedure described in Example 1. Any instrumentality other than this specific apparatus may be employed that effects the substantial freedom from contamination of the elemental constituents and the resulting alloy and that facilitates the diffusion of the constituents with one another. The mixture is then heated for approximately twenty hours at a temperature below the fusion point of the lowest melting constituent and the resulting alloy but sufficiently high to enable the constituents to diffuse into each other. The temperature range found satisfactory for this alloy is between 800° and 1200° C., a temperature of 900° C. being desirable. The resultant product after cooling is removed from the container and is rather easily broken in an attrition mill or other comminution means. This alloy as well as others, such as iron-nickel-molybdenum, iron-nickel-chromium, and iron-nickel-cobalt-molybdenum prepared by this method when placed in dust cores exhibits magnetic and electrical characteristics substantially similar to the corresponding alloys comprising the same quantity of constituents and prepared by the fusion method. This method may be applied to the production of other magnetic alloys, such as iron-nickel-tungsten, iron-cobalt-molybdenum, iron-cobalt-nickel-manganese, iron-nickel-silicon and the binary and ternary alloys of iron, nickel and cobalt.

Intermediate steps not appearing in the specific examples described may also be employed without materially diverting from the scope of this invention.

What is claimed is:

1. A method of producing a magnetic material in finely divided form which comprises mixing a metal and an element in the desired proportions, said mental and said element being in a finely divided form, and heating said mixture at a temperature below the fusion point of said metal and said element and sufficiently high to effect the substantial diffusion of said element and said metal.

2. A method of producing a magnetic material in finely divided form in accordance with claim 1, in which the metal comprises iron and nickel and the element is silicon.

3. A method of producing a magnetic material in finely divided form in accordance with claim 1, in which the metal comprises iron and the element is silicon.

4. A method of producing a magnetic material in finely divided form which comprises mixing a plurality of metals, said metals being in a finely divided form and heating said mixture at a temperature below the fusion points of said metals and sufficiently high to effect the substantial diffusion of said metals.

5. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of the metals comprises nickel and iron and another a member of the chromium-molybdenum-tungsten group.

6. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of the metals comprises nickel and iron and another comprises manganese.

7. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of said metals comprises cobalt and another comprises iron.

8. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of said metals comprises iron, another nickel and another a member of the chromium-molybdenum-tungsten group.

9. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of said metals comprises iron, another nickel and another manganese.

10. A method of producing a magnetic material in a finely divided form in accordance with claim 4, in which one of said metals comprises iron, another cobalt and another a member of the chromium-molybdenum-tungsten group.

11. A method of producing a magnetic material in a finely divided form in accordance with claim 4, in which one of said metals comprises iron, another cobalt, and another manganese.

12. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of said metals comprises iron, another nickel and another cobalt.

13. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of said metals comprises iron and nickel and another comprises iron and cobalt.

14. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of said metals comprises iron and nickel, another comprises iron and cobalt and another a member of the iron-nickel-cobalt group.

15. A method of producing a magnetic material in finely divided form in accordance with claim 4, in which one of said metals comprises iron, another cobalt, another nickel, and another a member of the chromium-molybdenum-tungsten group.

16. A method of producing a magnetic material in finely divided form which comprises mixing an alloy and an elemental metal in the desired proportions, said alloy and

said metal being in a finely divided form, and heating said mixture at a temperature below the fusing point of said alloy and said metal and sufficiently high and for a period sufficiently long to effect substantial diffusion.

17. A method of producing a magnetic material in finely divided form which comprises mixing an alloy comprising iron and nickel and a metal of the tungsten-chromium-molybdenum group in the desired proportions, said alloy and said metal being in a finely divided form, and heating said mixture at a temperature below the fusing point of said alloy and said metal and sufficiently high and for a period sufficiently long to effect substantial diffusion.

18. A method of producing a magnetic material in finely divided form which comprises mixing a metal comprising manganese with an alloy comprising iron and nickel in the desired proportions, said metal and said alloy being in a finely divided form and heating said mixture at a temperature below the fusing point of said metal and said alloy and sufficiently high and for a period sufficiently long to effect substantial diffusion.

19. A method of producing a magnetic material in finely divided form which comprises reducing an alloy and an elemental metal to a finely divided form, mixing said alloy and said metal in the desired proportions, placing said mixture in a container, hermetically sealing said container, heating said mixture to a temperature below the fusion point of said alloy and said metal and sufficiently high to effect substantial diffusion, and reducing the resulting product to the desired finely divided form.

20. A method of producing a magnetic material in finely divided form in accordance with claim 19, in which the alloy comprises iron and nickel.

21. A method of producing a magnetic material in finely divided form in accordance with claim 19, in which the alloy comprises iron, nickel and cobalt.

22. A method of producing a magnetic material in finely divided form which comprises mixing an alloy comprising iron and nickel and a metal of the chromium-molybdenum-tungsten group in the desired proportions, both said alloy and said metal being sufficiently fine to pass through a 120-mesh sieve, placing said mixture in a container, hermetically sealing said container, heating said mixture to a temperature between 800° to 1200° C. for a period sufficient for substantial diffusion, and reducing the resulting product to the desired finely divided form.

23. A method of producing a magnetic material in finely divided form which comprises mixing an alloy comprising iron and nickel with manganese in the desired proportions, both said alloy and said manganese being sufficiently fine to pass through a 120-mesh sieve, and placing said mixture in a container, hermetically sealing said container, heating said mixture at a temperature between 800° to 1200° C. for a period sufficient for the substantial completion of diffusion, and reducing the resulting product to the desired finely divided form.

24. A method of producing a magnetic material in finely divided form which comprises mixing approximately 96 to 98 parts of an alloy comprising 75% to 85% nickel and the remainder chiefly iron with approximately 2 to 4 parts of a metal of the chromium-molybdenum-tungsten group, both said alloy and said metal being in a form which is sufficiently fine to pass through a 120-mesh sieve, placing said mixture in a container, hermetically sealing said container, heating said mixture at a temperature between 800° and 1200° C. for a period sufficient for the substantial completion of diffusion, and reducing the resulting sintered mass to the desired finely divided form.

25. A method of producing a magnetic material in finely divided form which comprises mixing approximately 96 to 98 parts of an alloy comprising 75% to 85% nickel and the remainder chiefly iron with approximately 2 to 4 parts of manganese, both said alloy and said manganese being in a form which is sufficiently fine to pass through a 120-mesh sieve, placing said mixture in a container, hermetically sealing said container, heating said mixture at a temperature between 800° and 1200° C. for a period sufficient for the substantial completion of diffusion, and reducing the resulting sintered mass to the desired finely divided form.

In witness whereof, we hereunto subscribe our names this 14th day of April, 1932.

EARLE E. SCHUMACHER.
WILLIAM C. ELLIS.