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2,937,964

MAGNETIC FLAKE CORE

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No Drawing. Filed July 23, 1957, Ser. No. 673,742

6 Claims. (Cl. 148—105)

(Granted under Title 35, U.S. Code (1952), sec. 266)

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

The present invention relates to the manufacture of magnetic cores, and more particularly to the manufacture of insulated flake-type magnetic cores in which insulated flat-flake particles, whose length and width are much larger than their thickness, of magnetic material of the nickel-iron alloy type are compressed to provide a high density magnetic core characterized by lower eddy current losses and higher permeability than nickel-iron magnetic core materials heretofore produced.

Magnetic cores for medium and high frequency uses are frequently made from powdered metallic magnetic materials. The most commonly used materials for the manufacture of powdered magnetic cores are Permalloy, Molybdenum-Permalloy, and 2-81 Molybdenum-Permalloy. The purpose of employing powdered magnetic materials is to provide increased stability against the effects of temperature and flux changes and to decrease the power losses due to eddy currents by incorporating air gaps between the magnetic powder particles. Although these air gaps are desirable in one respect, their presence is effective to produce deleterious effects in another respect, namely that the air gaps lower the effective magnetic permeability of the cores.

The quality of magnetic cores is generally assessed by the product μQ , where μ is the effective magnetic permeability of the core and Q is the ratio of the reactance of the core to the effective series resistance that the core adds to the circuit due to the power losses in the core. In the design of inductors for use at medium and high frequencies, it is generally desired to increase the effective magnetic permeability of the cores while simultaneously increasing the μQ factor or at least maintaining the μQ factor thereof constant. However, this condition is unattainable by present day powdered cores due to the fact that these cores are characterized by a relatively low effective magnetic permeability by virtue of their air gap structure and, in order to increase the permeability thereof, require a compromise between the selection of the effective permeability and the core power losses, thereby resulting in a selected permeability less than that desired and a μQ factor deviating from, and at a lower value than, the desired constant value.

The general purpose of this invention is to provide a magnetic core which embraces all of the advantages of prior art cores, of nickel-iron alloy composition and which overcomes the disadvantages inherent in such cores. A primary aim of the present invention is to improve the effective magnetic permeability of nickel-iron alloy cores while maintaining the product μQ either constant or at an increased value. The attainment of these objectives results in inductors of smaller physical size than equivalently rated prior art powder core inductors.

These objectives are accomplished, in accordance with the concept of the invention, by fabricating the cores

from layered flake particles of magnetic material instead of the equiaxed particles that are normally used in the manufacture of such cores. The layered flake particles provide for fewer air gaps in the direction of the magnetic flux while maintaining the air gaps in a direction normal to the magnetic flux. This condition results in a higher permeability with lower, or equally low, eddy current losses than an equivalent powdered magnetic core. The low eddy current loss coefficient coupled with the high permeability, as characterized by the magnetic material of the present invention, results in a magnetic flake core which is capable of covering the same frequency range as an equally-rated powder core with an increase of 100% in permeability thereover, the improved properties resulting in a physically smaller core size than equally-rated powder cores for any given application.

With the foregoing in mind, it is an object of the present invention to produce a magnetic material of nickel-iron alloy composition which is characterized by higher magnetic permeability than similar type magnetic materials heretofore produced.

Another object is to produce magnetic flake particles from magnetic powder particles.

A further object is to produce a magnetic core composed of insulated flake particles of nickel-iron composition compressed in parallel layers.

Yet another object is to produce a magnetic core composed of insulated flake particles of nickel-iron composition compressed in parallel layers such that fewer air gaps are present in the direction of magnetic flux than are present in a direction normal to the magnetic flux.

Still another object is the provision of a unique method for producing magnetic flake cores of basically nickel-iron composition having a magnetic permeability of substantially 100 percent higher than cores of similar material heretofore produced.

A more specific object is to form flake particles from powdered nickel-iron magnetic alloys and to process the flake particles in a novel manner so as to produce a magnetic core composed of flat insulated particles compressed in parallel layers.

An essential object of the invention is the process of forming a magnetic core composed of flat insulated particles, of basically nickel-iron composition, compressed in parallel layers, annealing the core in a hydrogen atmosphere followed by rapid cooling in air.

A principal object of the invention is the provision of a novel core-producing process which includes the steps of reducing a basically nickel-iron alloy into the form of powder particles, cold rolling the powder particles into flake particles, annealing the flake particles, insulating the particles, forming the flakes in a magnetically aligned compact, and annealing the compact followed by rapid cooling in air.

Still further objects and the entire scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, however, that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

In accordance with the present invention, it is contemplated to accomplish the foregoing objects by first forming magnetic flake particles from any one of several well known magnetic powders of the nickel-iron alloy type such, for example, as Permalloy, Molybdenum-Permalloy or 2-81 Molybdenum-Permalloy and then subjecting the flake particles to a process including the operations of (1) annealing the flakes to eliminate internal stresses resulting from the flake-forming operation and to im-

prove the ductility thereof so as to enhance compaction of the flakes into a suitably shaped bulk; (2) insulating the flakes with an inorganic binder; (3) aligning the flake particles in parallel planes which are parallel to the direction of the magnetic flux path of the core; (4) compacting the flakes into a solid mass of a configuration suitable for the desired application; (5) annealing the mass to relieve strains resulting from the compacting operation; (6) and rapidly cooling the annealed mass in air.

The initial flaking operation is performed by cold rolling magnetic powder particles so as to form flake particles which have a particle-diameter three to twenty times the thickness of the particles, the desired diameter size being determined by sieving the flake particles. The process of this invention may use commercially available powder particles, of the nickel-alloy type, as the initial product, or may use powder particles produced in the manner hereinafter described. Although the process, as briefly set forth above is hereinafter described with particular reference to the production of a core, it is to be understood that the process may be terminated after the flake-forming operation, and the flake particles used to fabricate magnetic products utilizable for various electrical applications. Alternatively, the final bulk product obtained by the hereinabove outlined process may be worked into any desired shape or form suitable for an intended magnetic application.

The success of the invention is due to several factors. It is essential that the flakes have a particle-diameter size at least thrice the thickness thereof. In this manner, comparatively fewer air gaps are present in the direction of the magnetic flux than exist in a direction normal to the magnetic flux. Otherwise, the magnetic and electrical characteristics will be generally uniform irrespective of the direction of magnetic flux, resulting in the nullification of the advantages derived from the flake configuration.

It is also essential to the success of the hereinabove described process that the compacted bulk be rapidly air-cooled after the annealing operation. This rapid cooling step at this stage of the material is unconventional and contrary to established practice in the final treatment of nickel-iron alloys of this type. However, it has been found that the rapid cooling treatment results in an unexpected increase in core permeability and reduction in core losses.

The invention is illustrated, but not limited, by the following specific example of the preparation of a magnetic flake material of preferred composition. Wherever possible, alternate modes of operation are discussed, but it will be recognized that various additional modifications can be made without deviating from the scope of the invention.

As aforesaid, the starting product, upon which the process of the invention may be practiced to produce magnetic flake cores, may be any one of several commercially available nickel-iron powder alloys or may be any of the powder-particle alloys described in U.S. Patents 1,669,649 and 1,859,067 to Beath et al., and 1,878,589 to Maris et al., or as described in the article by Shackleton and Barber in the Transactions of the American Institute of Electrical Engineers, vol. 47, 1928. Alternatively, the powder particles are preferably produced in the novel manner described below which provides 2-31 Molybdenum-Permalloy powder particles. It is to be understood that it is highly desirable to use very fine-equiaxed grain-size powder particles in order to produce a magnetic flake core of optimum quality, the hereinbelow powder forming operation being practiced in a manner to attain this end.

Forming magnetic powder

A melt is prepared by melting high purity nickel, iron and molybdenum, under vacuum in a suitable furnace, to form a molten alloy in which the proportions of the

constituents are nominally 2% by weight of molybdenum, 81% by weight of nickel, and the balance essentially iron.

Although an alloy of the above prescribed ingredients is somewhat brittle, it is desirable to further embrittle the alloy so as to assure optimum separation of the grains into fine powder particles. To further embrittle the alloy, sufficient sulfur or sulfur compound may be added to the molten alloy so as to provide a sulfur content of 0.02% by weight of the total alloy. It is to be understood, however, that the addition of sulfur is optional and serves only to enhance the brittle characteristic of the alloy. If it is desired, the melt may be prepared in an air atmosphere instead of in a vacuum, in which event sulfur is not necessary as an embrittling agent. The utilization of sulfur as an embrittling agent in nickel-iron alloys is fully described in U.S. Patent 1,739,052 to White and may be employed in the process of the present invention in the manner set forth by White.

After the ingredients of the melt have been thoroughly commixed, the melt is cast into an ingot or slab, preferably the latter. The casting is permitted to cool to the point where it is sufficiently solid, either while still red hot or cold, to be removed from the mold, whereupon it is placed in a furnace and heated at a temperature between 1150° C. to 1300° C., but preferably at 1250° C., for a period of time sufficient to uniformly heat the slab, the specified temperature range being substantially above the recrystallization temperature of the material. Alternatively, the red hot slab may be directly rolled without reheating if it is within the prescribed temperature range.

The hot slab is then passed, preferably without subsequent reheating, through progressively reduced rolls at such a rate that the slab has been reduced to a sheet of predetermined thickness such, for example, as 0.2 inch at about the time that the material has cooled to a temperature slightly above that at which the material ceases to be malleable. This hot rolling operation is effective to produce a large-grained crystalline structure of equiaxed configuration in the material.

Should the material become cooled while passing through the rolls to a temperature slightly above that at which it ceases to be malleable, the rolling operation may be temporarily terminated and the material reheated to a temperature at which it becomes malleable, the rolling operation being then continued to the final desired thickness.

The hot sheet is then immediately quenched in water to disorder the atomic structure of the material for the purpose of making the sheet malleable under cold-rolling conditions. The sheet is thereafter sand-blasted or cleaned in any conventional manner to remove the loosely adhering oxide coating.

At this point, the product is a sheet of large-equiaxed grain structure. In order to obtain fine powder particles, it is necessary to reduce the size of the grains. This is accomplished by sufficiently cold working the material so as to result in a fine-equiaxed grain structure upon the material being subjected to a recrystallization treatment.

The cold working of the material breaks down the large-grained crystalline structure of the hot-rolled slab and produces, upon being heat-treated above the recrystallization temperature, a fine-equiaxed grain product which is extremely brittle. If a small amount of sulfur has been previously added to the material in the manner aforesaid, fracture of the material occurs principally along the crystal boundaries, due to the incorporated sulfur being collected between the crystals of the material.

The cold working is accomplished by passing the slab, at room temperature or at between 20° C. to 40° C. which is below the recrystallization temperature, through rolls the spacing between which is progressively de-

creased for successive passes to produce a thinner sheet, preferably of a thickness between 0.1 to 0.15 inch.

Thereafter, the sheet is subjected to a recrystallization heat-treatment by heating it in an air atmosphere at just above the recrystallization temperature for one hour, or by heating at a slightly higher temperature for a shorter period of time. The recrystallization heat-treatment is effective to greatly reduce the size of the crystal grains so that the resultant sheet is characterized by a fine-equiaxed grain structure.

Since the recrystallization temperature of any given material is dependent upon numerous factors, predominantly the proportioning of the ingredients and the amount of cold working, it is necessary to determine the recrystallization temperature of the material prior to the recrystallization treatment. This may be conveniently achieved by subjecting a plurality of specimens from the material to different temperatures for a period of one hour, and microscopically determining at which temperature recrystallization occurs. Upon making this determination, the aforescribed recrystallization treatment is performed.

The sheet material obtained by the above operations is then fractured and crushed in a jaw crusher, hammer mill, or any other suitable type of apparatus, after which the crushed material is rolled in a ball mill until it is reduced to a fine powder. The powder is then sieved through 200 mesh sieve, the ultimate product passing through the sieve being used in the operations subsequently to be described and being fine powder particles characterized by an equiaxed configuration.

Although the operations subsequently to be described are of specific application to the powder-particle product produced in the manner aforescribed, the subsequent operations are also effectively applicable to commercially available magnetic powder of nickel-iron alloys or to the powder alloys produced by the aforementioned patents.

Flaking

The powder particles are flattened by cold rolling, at room temperature or 20° C. to 40° C., in a laboratory two-high rolling mill to produce flakes whose average diameter is at least three times as great as their thickness. A single pass of the particles through the rolling mill is generally sufficient to produce the desired flakes.

Annealing

In order to remove internal strains introduced by the cold-rolling operation and to improve the ductility of the flakes for compaction purposes, the flakes are heat treated in a hydrogen atmosphere at a temperature sufficient to cause annealing and yet low enough to prevent sintering. In addition, the hydrogen is effective to eliminate the presence of sulfur from the flake-mass by reacting therewith, the presence of sulfur in the flake-mass no longer being desired since it has served its purpose in the embrittling operation. A suitable range for this operation is 600° C. to 850° C. for periods of 3 to 1/2 hour, respectively. The annealing operation, though, is preferably carried out at a temperature of 750° C. for a period of one hour.

The flakes are then permitted to slowly cool to room temperature in the hydrogen atmosphere to avoid oxidation and the re-introduction of strains.

Insulating

An insulating film is required on each particle before compacting to reduce the effect of eddy current losses. This film must be as thin as possible to provide a high core permeability and possess enough strength to withstand the extreme pressures and abrasions of pressing. A ceramic type of insulation which is found suitable for the purpose is similar to that described in U.S. Patent 2,230,228 to Bandur, although differing therefrom sub-

stantially in the manner of application and in ingredient composition and proportioning.

The flakes are insulated by applying either 3 or 4 layers of insulant in sufficient quantity to constitute approximately 3% to 4% of the total weight of the alloy.

A typical example of the composition of each layer in the 4 layer process is as follows:

	Material	Percent of flake weight
10		
First layer	Talc.....	0.400
	Mg(OH) ₂	0.027
	(Na ₂ SiO ₃).....	0.173
15	Second layer	
	Talc.....	0.280
	Mg(OH) ₂	0.021
	(Na ₂ SiO ₃).....	0.099
Third layer	Kaolin.....	0.5000
	Mg(OH) ₂	0.1875
Fourth layer	(Na ₂ SiO ₃).....	0.3125
	(¹).....	(¹)

20 ¹ Same as third layer.

The total composition of the above four layers is 3.0% by weight of the total alloy. If it is desired to insulate the particles in only three layers, the first two layers may be applied as per above and the percentage contents of ingredients in the third layer doubled. If it is desired to provide an insulant constituting about 4% of the total weight of the alloy, the first two layers are applied as per the above table and the compositional contents of the third layer tripled, no fourth layer being applied.

Returning now to the manner of applying the insulant as per the hereinbefore described table, the talc, magnesium hydroxide, and the sodium silicate are combined with enough water to make an easily flowing slurry. The slurry is commixed with the flakes in a rotating tumbler which is being simultaneously heated to a temperature between 80 to 120° C. to cause evaporation of the water. After the water has evaporated, the agglomerated particles, which tend to adhere together, are separated in any suitable manner such, for example, as by agitation, vibration, or by soft rubber rolls.

This process is repeated with the next layer of insulant until all four layers have been applied. It is to be noted that this method of insulant-application substantially differs from that of the aforementioned Bandur patent.

Aligning flakes

In order to obtain optimum benefits from the insulated flakes, it is desirable to layer them in parallel planes with the flat faces parallel to the direction of magnetic flux so that fewer air gaps are present in planes parallel to the magnetic flux than exist in the plane normal to the magnetic flux.

The aligning of the insulated flakes may be accomplished by allowing them to fall freely into a die from a rotating funnel which is positioned over the die. On the other hand, flake alignment may be obtained in any conventional manner such as vibration or magnetic alignment.

Compaction

After the flakes are layered in the die, they are compacted under a pressure of 80 to 120 tons per square inch, optimum results having been attained at a pressure of 100 tons per square inch. The compaction operation of the flakes is effective to produce a strong, dense core structure.

Annealing

The core is then annealed in a hydrogen atmosphere at temperatures between 600° C. to 700° C. in a furnace for a short period of time to eliminate the strains caused during the compaction operation. Care must be exercised in not heating the core for too long a period of time, otherwise the interparticle insulation is destroyed with an attendant increase in eddy current losses. The

most suitable conditions for this operation were found to be 650° C. for 20 minutes.

Rapid cooling

Upon completion of the annealing operation, the core is immediately removed from the furnace and rapidly cooled in air. This rapid cooling operation is contrary to the established practice for the final treatment of molybdenum, nickel-iron alloys. However, this unconventional treatment produced the unexpected results of decreasing the core losses and enhancing the permeability properties thereof.

If desired, the core may be enameled with any type of organic high-dielectric material commonly used for this purpose to give added strength and to prevent absorption of moisture. In lieu of an organic coating, the core may be lacquered with varnish.

The resultant core obtained from the above procedures is characterized by lower eddy current losses and higher permeability than equally rated nickel-iron powder cores. Also, for designs of equivalent electrical characteristics, the core material of the present invention is of smaller size than an electrically similar nickel-iron powder core.

Obviously, many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood that, within the light of the teachings herein and the appended claims, the invention may be practiced otherwise than as specifically described.

What is claimed as new and described to be secured by Letters Patent of the United States is:

1. The method of making a magnetic core characterized by a high magnetic permeability which comprises the process of forming a magnetic core composed of flat insulated particles, of an alloy containing about 2% molybdenum the remainder essentially nickel-iron, compressed in substantially parallel layers; annealing the core in a hydrogen atmosphere; and thereafter immediately subjecting the core to an air atmosphere for rapid cooling in air.

2. The method of producing a magnetic core which includes the steps of reducing a molybdenum, nickel-iron alloy containing at least about 2% molybdenum into the form of powder particles, cold rolling the powder particles at substantially room temperature to form flake particles therefrom, annealing the flake particles, insulating the particles with an inorganic insulant, forming the particles in a magnetically aligned compact, annealing the compact in a hydrogen atmosphere at a temperature between 600° C. to 700° C. for a period of time at least to uniformly heat throughout, and thereafter immediately quenching the compact in air.

3. The method of claim 2, wherein said cold rolling is carried out at temperatures between 20° C. to 40° C., the annealing of the compact is performed in a hydrogen atmosphere at a temperature of approximately 650° C. for a period of about 20 minutes, and the percentage content of said inorganic insulant constituting about 3% by weight of the flake particles.

4. The method of producing magnetic cores which includes the steps of reducing a molybdenum, nickel-iron alloy containing at least about 2% molybdenum into the form of powder particles, rolling said particles at temperatures between 20° C. to 40° C. to form therefrom

flake particles, annealing the flake particles by heating in a hydrogen atmosphere at a temperature of 750° C. for a period of one hour, slowly cooling the particles in a hydrogen atmosphere to room temperature, insulating the particles with an inorganic insulant applied in several discrete layers, forming the particles in a magnetically aligned compact, annealing the compact by heating in a hydrogen atmosphere at a temperature of 650° C. for 20 minutes, and thereafter immediately quenching the compact in air.

5. The method of making magnetic cores of flaked particles comprising the steps of forming a melt consisting of 2% molybdenum, 81% nickel, and the balance essentially iron, casting the melt into a slab, progressively reducing the thickness of the slab by working above the recrystallization temperature for a predetermined interval followed by working below the recrystallization temperature, comminuting the reduced slab to form fine powder particles, cold working the powdered particles at substantially room temperature to form flake particles, heat treating the flake particles in a hydrogen atmosphere at a temperature sufficient to anneal but insufficient to sinter the flake particles, insulating the flake particles with an inorganic insulant, aligning the flake particles in parallel planes, compacting the particles under pressure into a suitable shape, annealing the compacted particles in a hydrogen atmosphere, and thereafter immediately subjecting the compacted particles to an air atmosphere for rapid cooling to room temperature.

6. The method of making magnetic cores of flaked particles comprising the steps of forming a slab of an alloy consisting of 2% molybdenum, 81% nickel, and the balance essentially iron, progressively reducing the thickness of the slab by working above the recrystallization temperature for a predetermined interval followed by working below the recrystallization temperature, comminuting the reduced slab to form fine powder particles, cold working the powdered particles at room temperature to form flake particles, heat treating the flake particles at a temperature of 750° C. for an interval of one hour in a hydrogen atmosphere, coating the flake particles with discrete layers of an insulant, aligning the flake particles in parallel planes, compacting the particles under pressure into a suitable shape, annealing the compacted particles at a temperature of 650° C. for 20 minutes in a hydrogen atmosphere, and thereafter immediately subjecting the compacted particles to an air atmosphere for rapid cooling to room temperature.

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