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METHOD OF MAKING A MAGNETIC SHEET

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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.

This invention relates to a method of making a sintered magnetic alloy having high permeability, high resistivity, very low magnetostriction and magnetic anisotropy, and more specifically, to a method by which it is possible to manufacture said alloy in the form of thin sheets.

A high permeability, low hysteresis-loss family of soft magnetic alloys, commonly known as Sendust, is described in U.S. Patent 2,193,768 to Masumoto et al. Certain compositions within this alloy system are known to possess such desirable characteristics as low magnetostriction and magnetic anisotropy, high initial and maximum permeability, and high resistivity. However, the alloys within this system are known to be very brittle and cannot be readily machined or forged into the desired shapes. They must either be melted and cast by conventional foundry techniques into the shapes desired or they must be powdered and pressed into desired configurations using conventional powder metallurgy techniques. Thin laminations or close tolerance sections cannot be made by normal melting and casting procedures because of warping and cracking that takes place during the cooling cycle. Therefore Sendust produced by normal foundry techniques is unsuitable for use in A.C. power applications because of the high eddy current losses associated with unlaminated cast configurations. Insulated pressed powder cores eliminate this eddy current problem and are suitable for high frequency inductor use but are not suitable for A.C. power applications because of their low permeabilities and low magnetic saturation induction values.

It is therefore an object of this invention to provide a sintered alloy of the Sendust type which may be readily formed into thin sheets and stamped into desired configurations.

Another object of the present invention is the provision of a method of making a ternary magnetic alloy containing iron, aluminum and silicon having superior magnetic and physical properties and to which additives may be selectively introduced to alter the physical and magnetic properties of the alloy.

A further object of the invention is the provision of a method of making a sintered alloy of the Sendust type which may be formed into thin sheets and cut into shapes suitable for use in high frequency A.C. applications without suffering large eddy current losses.

In practicing this invention, the composition of the alloys manufactured thereby lie in the range of 6 to 12% silicon, 4 to 9% aluminum, and the remainder being iron. If desired, small amounts of additives such as antimony, beryllium, nickel, chromium, tungsten, molybdenum, magnesium, manganese, vanadium, tantalum, titanium, tin, zinc, boron, copper, phosphorus, arsenic, sulfur or zirconium may be introduced to the alloy to selectively modify the physical and magnetic properties of the alloy, such as

resistivity, hardness, permeability or coercive force. It has been found that effective resistivity controlling additives are P in the form of FeP where the P is present in amounts up to 0.25% of the final alloy or Sn in an amount up to 3% of the final alloy.

In the practice of this invention, the Sendust alloy is made by mixing an iron deficient master alloy of the Sendust type alloy with iron in the form of a powder or flake and treating the mixture in a method to be described.

The degree of iron deficiency in the master alloy may be selectively varied to control the physical and magnetic properties of the final alloy. The master alloy is a Sendust type alloy and is prepared by conventional melting and casting of elemental aluminum, silicon and iron, said iron being present in an amount approximately 75%, for example, of a predetermined total amount to be present in the final alloy. Because of this iron deficiency the resulting master alloy is extremely brittle and is then easily pulverized to a powder by means of a jaw mill, disc crusher, and ball milling operation.

The powdered master alloy, having a 25% iron deficiency, is then mixed with elemental iron in either powder or flake form in a quantity sufficient to correct the 25% iron deficiency of the master alloy. The addition of elemental iron to the powdered master alloy permits precise control over the final ratio of silicon plus aluminum to iron in the final Sendust alloy. The powdered alloy and the iron powder are thoroughly mixed and then directed into a standard two-high powder rolling mill to compact the powder mixture and form said mixture into a green strip. The ductile iron powder in the powdered master alloy serves as an excellent bonding agent for the hard, brittle, iron deficient Sendust particles which otherwise could not be compacted and rolled into a green strip having sufficient ductility and green strength to facilitate handling. Additional strength may be imparted to the compacted powder strip by a short bonding anneal in a pure dry inert atmosphere such as argon and helium gases. The temperature used for this bonding operation may range from 600° C. to 1300° C. for periods from three hours to five minutes, these variables being controlled so as not to effect complete diffusion and consequent embrittlement of the strip. Following the short bonding anneal for additional strength, the strip is then cold-rolled on a standard two-high rolling mill to the final thickness desired. This second cold-rolling operation may be used to achieve reductions of thickness of the strip as great as 50%, with the final thickness being dependent upon the thickness of the green strip emerging from the first powder-rolling operation.

The sheets of Sendust alloy emerging from the second rolling mill are then stamped in a stamping die or cut into desired shapes for use in the final magnetic core to be produced. These laminations are then subjected to a final sintering operation in an inert atmosphere wherein temperatures in the range of 1,000° C. to 1400° C. are used for periods of time from 1 hour to 16 hours. During this final sintering operation, the Sendust type material experiences complete and uniform diffusion of the constituents into a homogeneous Sendust alloy. The exact sintering temperature which is used is dependent upon the type and size of iron powder used to correct the iron deficiency and is also effected by the presence of certain previously mentioned additives for modifying the physical and magnetic properties of the alloy. The duration of the

final sintering operation is generally inversely proportional to the temperature applied. No additional heat treatments are required to produce the optimum magnetic properties in the material.

After the iron deficient master alloy has been pulverized and mixed with free iron to correct the iron deficiency and the powder mixture has been compacted in the first rolling mill to form a green strip, the green strip could then be subjected directly to the final sintering operation and thereby bypass the presintering step, the second cold rolling step, and the stamping operation, all of which are considered to be optional steps in the performance of the method of this invention. That is to say, it is considered to be within the scope of this invention that the Sendust alloy may be made by eliminating one or more of the presintering, second cold rolling, or stamping operations, dependent upon the properties and configurations desired in the final product.

In practising this invention, and using 9.5% silicon, 5.7% aluminum and 84.8% iron as the constituents in the final Sendust alloy, it has been possible to produce Sendust strips having thicknesses as low as 10 mil, and further reductions in thickness are feasible using slight modifications in rolling equipment and by varying the particle size of the powders. Moreover, when using ring laminations stamped from a Sendust sheet of 0.014 inch thickness, and produced by the method of this invention, the following D.C. properties are exhibited by the ring:

μ_{20}=16,130; μ_{max}=36,625; H_c=0.062 oersted B_r=3,450 gauss; B_m=8,960 gauss wherein μ_{20} is the initial permeability at 20 gauss, μ_{max} is the maximum permeability, H_c is the coercive force, B_r is the remanence, and B_m is the saturation level. The Sendust ring lamination of 0.014 inch thickness also exhibited a lower core loss at high frequency A.C. operation, i.e. below 1 magacycle, than a lamination of molybdenum permalloy of the same thickness. At the higher frequencies, resistivity becomes more important than permeability in the example cited, Sendust resistivity is 106 micro ohm-cm. as compared with 55 micro ohm-cm. for Mo-Permalloy. Furthermore, at 1.25 mc./sec. a permeability of 71 was obtained on a 0.011" thick Sendust lamination as compared to a permeability of 46 for a Mo-Permalloy lamination of the same thickness. The foregoing magnetic characteristics of the Sendust alloy produced by the method of this invention is evidence that said alloy is ideally suited for extensive and varied applications. The addition of free iron powder or flakes to correct the iron deficiency in the powdered master alloy not only contributes greater ductility and therefore better physical properties to the final Sendust alloy but also serves to improve the magnetic characteristics of the alloy. By improving the magnetic characteristics of the Sendust alloy as well as the physical properties thereof, the method of the present invention makes possible the efficient use of Sendust alloys as magnetic cores in A.C. power applications as well as in D.C. applications.

What is claimed is:

1. The method of making a ternary magnetic alloy containing about 4–9% Al, 6–12% Si, and the remainder essentially Fe comprising the steps of
- pulverizing into a powder a master alloy containing the total predetermined amounts of Al and Si desired in the final alloy and only 75% of the total predetermined amount of Fe desired in the final alloy,
- adding Fe powder to the powdered master alloy in an amount which, when added to the amount of Fe present in the powdered master alloy, equals a predetermined amount of Fe desired in the final alloy, and thoroughly mixing said powders,
- compacting or rolling the powdered mixture into a sheet, and
- sintering said sheet at a temperature of 1000 C. to 1400° C. for 1 to 16 hours in an inert gas atmosphere.

2. The method of claim 1 wherein the powder mixture contains 9.5% Si, 5.7% Al, and 84.8% Fe.

3. The method of claim 1 wherein the sintering operation is conducted at 1300° C. for 4 hours, and the inert gas atmosphere is argon.

4. The method of forming a sintered compact containing about 4–9% Al, 6–12% Si, the remainder essentially Fe, which comprises the steps of
- casting an ingot of a master alloy having the aforementioned elements, said Fe being present in an amount approximately 75% of the final amount desired,
- grinding the master alloy ingot into a powder,
- adding sufficient Fe powder to the powdered master alloy until the total Fe present is equal to the final amount desired in the sintered compact,
- compacting the powder mixture in a powder rolling mill to form a green strip,
- sintering the green strip in an inert atmosphere at a temperature of 600° C. to 1300° C. for a period of 5 minutes to 3 hours to improve the strength of the strip,
- cold rolling the strip by means of a rolling mill to the desired thickness, and
- sintering the strip in an inert atmosphere at a temperature of 1000° C. to 1400° C. for 1 to 16 hours.

5. The method of claim 4 wherein
- the sintered compact contains 9.5% Si, 5.7% Al, and 84.8% Fe.

6. The method of claim 5 wherein
- the first mentioned sintering operation is conducted at 1200° C. for 35 minutes, and
- the second mentioned sintering operation is conducted at 1300° C. for 4 hours.

7. The process of forming a sintered magnetic alloy containing 4–9% Al, 6–12% Si, and the remainder essentially Fe comprising the steps of
- pulverizing a ternary master alloy containing the total predetermined amounts of Al and Si desired in the final alloy but only 75% of the predetermined amount of Fe desired in the final alloy,
- adding elemental Fe powder to the master alloy and additive mixture in a quantity so that the resultant mixture contains a predetermined amount of Fe desired in the final alloy,
- compacting or rolling the powder mixture into a sheet,
- heating said sheet in an inert atmosphere at a temperature in the range of 600° C. to 1300° C. for a period of time ranging from 5 minutes to 3 hours,
- cold rolling the sheet to the desired final thickness,
- cutting the sheet into shapes for use in laminated cores,
- heating the laminations in an inert atmosphere at a temperature between 1000° C. and 1400° C. for a period of time between 1 and 16 hours,
- whereby a sintered alloy is produced which may be efficiently used in A.C. power applications at frequencies as high as 1.25 mc./sec.

8. The process of claim 7 wherein
- the Al is present in an amount constituting 5.7% of the final alloy, and
- Si is present in an amount constituting 9.5% of the final alloy.

9. The process of claim 8 including the step of adding a resistivity controlling additive to the pulverized master alloy wherein the resistivity controlling additive is P in the form of FeP, said P being present in amounts up to 0.25% of the final alloy.

10. The process of claim 8 including the step of adding a resistivity controlling additive to the pulverized master alloy wherein the resistivity controlling additive is Sn and is present in an amount up to 3% of the final alloy.

11. The process of claim 9 wherein
- the first mentioned heating step is conducted at 1200° C. for 35 minutes, and

the second mentioned heating step is conducted at 1300° C. for 4 hours.

12. The process of claim 10 wherein the first mentioned heating step is conducted at 1200° C. for 35 minutes, and the second mentioned heating step is conducted at 1260° C. for 4 hours.

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