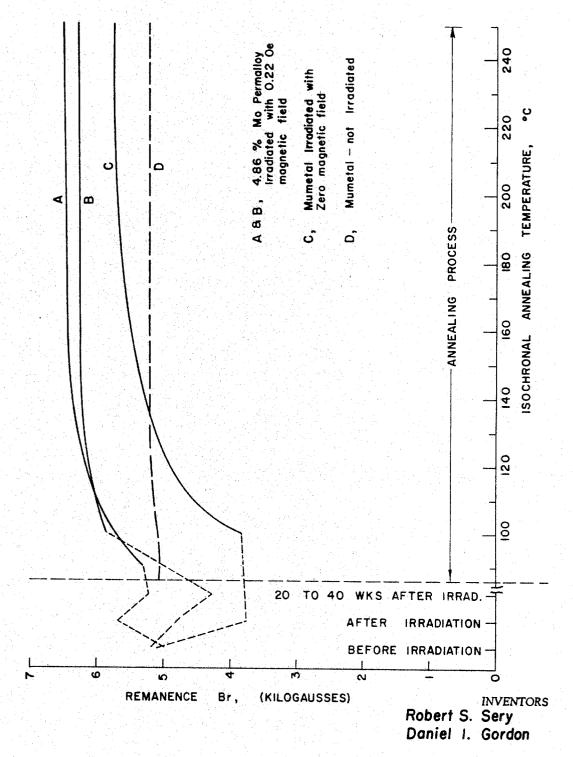
R.S. SERY ET AL
METHOD OF PRODUCING HIGH RECTANGULARITY, LOW
COERCIVE FORCE MAGNETIC CORES
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BY

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3,477,883 METHOD OF PRODUCING HIGH RECTANGULAR-ITY, LOW COERCIVE FORCE MAGNETIC CORES Robert S. Sery, Silver Spring, and Daniel I. Gordon, Chevy Chase, Md., assignors to the United States of America as represented by the Secretary of the Navy Filed Feb. 4, 1966, Ser. No. 525,812 Int. Cl. H01f 1/02

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2 Claims

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ABSTRACT OF THE DISCLOSURE

A method of producing magnetic cores having highly rectangular hysteresis loops and low coercive force by irradiating the core material in a low temperature environa low temperature.

The invention described herein may be manufactured and used by or for the Government of the United States 20 of America for governmental purposes without the payment of any royalties thereon or therefor.

This invention relates to a method for treating materials of high magnetic permeability to impart to them the characteristic of a rectangular hysteresis loop.

Magnetic materials having rectangular hysteresis loops have extensive application in present day technology, as in magnetic memory devices, computers, magnetic amplifiers and in instrument and distribution transformers. Magnetic materials having this property have been produced in the past either by applying a magnetic field to the material during a high temperature annealing process, by grain orientation obtained by drastic cold rolling reduction prior to final annealing, or by irradiating the material in the presence of a magnetic field with neutrons 35 or electrons. The first two of the aforementioned methods require high temperatures of 600° C. or greater and very long annealing times (18 hours or greater). The method of neutron irradiation in the presence of a magnetic field requires a costly nuclear reactor as a neutron source. While neutron irradiation will produce the desired rectangular hysteresis characteristic it also undesirably increases the coercive force (Hc) and involves long irradiation times; moreover the material becomes radioactive. In the method of electron irradiation an externally applied field is essential during the period of irradiation which is a restriction for some conditions or geometrical configurations.

The present invention produces magnetic materials having rectangular hysteresis loops, high values of remanence (B_r) maximum permeability (μmax.), and low coercive force (H_c) by a two-step method consisting of irradiating magnetic cores with electrons with or without an externally applied magnetic field within low temperature surroundings and then heating the cores in a furnace in the presence of an externally applied magnetic field. The above noted disadvantages encountered in the prior art are thus overcome such that the use of high temperatures or the use of magnetic fields during irradiation is eliminated.

It is an object of this invention to provide a new method of producing a magnetic core having improved magnetic properties.

Another object of the invention is to provide a new method of producing a magnetic core having improved magnetic properties without using the high temperatures associated with magnetic annealing.

A further object of the invention is to provide a new method of producing a magnetic core having hysteresis loops of high rectangularity and low coercive force.

Still another object of the invention is to provide a simple two-step method of producing a magnetic core

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having a rectangular hysteresis loop which can be carried out under more precise control conditions.

Other objects, advantages and novel features of the invention will become apparent from the following detailed description of the invention when considered in conjunction with the accompanying drawing wherein a graph is shown displaying the change in remanence of various samples of magnetic cores under precisely controlled irradiation and magnetic annealing conditions.

In this invention hysteresis loops of high rectangularity, high values of remanence, maximum permeability and low coercive force are obtained by a two step process. This process consists of irradiating magnetic cores with an electron beam from a suitable electron source, such ment, then magnetically annealing the material also at 15 as a Van de Graaff accelerator with or without the application of an externally applied magnetic field in the presence of temperatures less than 100° C. This irradiation step is followed by heating the cores in a furnace for approximately one hour at some temperature between 100° and 250° C. with an externally applied magnetic field present.

> The two step process allows better control of the production of the desired magnetic properties since the second step is done directly (not by remote control) whereas the first step, in common with all the irradiation methods, is done remotely. Because, in this process, the actual squaring up of the hysteresis loops (i.e. attainment of high rectangularity, remanence and maximum permeability) is accomplished in the second step of the two step treatment, the temperature is much more easily controlled. Moreover, since the temperature of the post-irradiation magnetic-annealing treatment and the time of its application are more amenable to control, the coercive force can be maintained at a low value. The reason for this is that certain types of defects, more specifically, vacancies in a crystalline lattice, can be placed in a material by several methods, such as quenching from sufficiently high temperature, e.g. 600° C. or greater, mechanical deformation of a material as by bending or by irradiation by atomic particles. However, it is the subsequent history of the temperature treatment which determines the extent and manner of the diffusion of these defects through the lattice. The number of these defects and the way in which they are distributed in the lattice determines the direction (+ or -) and magnitude of the changes in the properties of the material. Therefore, if the defects can be introduced in a first step as in this invention, then their diffusion can be accurately controlled in a second step. The desired property changes can be more easily attained in the two step process than in the one step process such as previous irradiation treatments or than in the thermomagnetic type processes (e.g. a preliminary anneal to relieve strains produced by the fabrication process followed by a separate magnetic anneal, both of approximately 16 to 24 hours duration).

> The magnetic field need not be present during the irradiation step. By the method of this invention a relatively large number of samples could be processed without the necessity, as in previous irradiation processes, of providing a separate current lead for each sample to produce the required applied magnetic field. The low-temperature post-irradiation magnetic-anneal treatment would bring about the desired property changes. This lack of an applied magnetic field during irradiation would also be advantageous when irradiating samples which require large magnetic fields for magnetic annealing. Such fields might be difficult or inconvenient to apply during irradiation but could be more easily applied during the second step of this method. Such samples include strips, C shaped or E shaped cores or any such open circuit sample configuration.

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By way of example several samples of polycrystalline 4.86% Mo Permalloy and Mumetal were treated according to the method of this invention in the following manner; punched rings having outer diameters of 13/16 inch and inner diameters of 5/8 inch and thicknesses of 0.010 and 0.021 inch respectively were irradiated by a 2 m.e.v. electron beam from a Van de Graaff acelerator. The beam was incident on the upper face of a hollow aluminum target block through which CO2 coolant gas passed. The samples rested in shallow circular wells in 10 the face of the block. For each of several samples a toroidal magnetic field was generated within this sample by means of a direct current in a wire fastened to the target block through the center of the sample. One sample was irradiated with no externally applied field present. Temperatures during irradiation were maintained within the range of 50° to 95° C. The electron flux and total dose were approximately 1.5×1013 electrons/ cm.2·sec. and 1×1017 electrons/cm.2 respectively. After this treatment these samples were placed in a furnace and held at 150° C. for one hour with an applied magnetic field of 0.22 Oe present. They were then air cooled and tested at room temperature at about 25° C. These samples, which had shown only slight changes in properties after irradiation showed large increases in rectangularity, remanence and maximum permeability with only slight or negligible changes in coercive force after this low temperature magnetic anneal.

Curves A, B, C and D of the attached drawing depict a characteristic change in remanence (B_r) of different 30 ings. core samples under particular conditions corresponding to the two step process of the immediate invention.

Two irradiation experiments, with a magnetic field applied, on 4.86% Mo Permalloy and Mumetal respectively disclosed that, although the total electron dose was achieved as in previous experiments the temperatures were significantly lower than in the previous ones. Moreover the magnetic property changes were less than had been expected from previous experimental work; in some cases the changes were even in a negative direction, e.g. remanence B_r decreased in one sample instead of increasing. Curve B of the attached drawing depicts such a decrease in remanence during the irradiation step.

Curves A and B were derived from two respective samples of 4.86% Mo Permalloy which were irridiated and annealed in acordance with the process of the instant invention. The isochronal annealing process is carried out by heating the samples in the presence of an externally applied magnetic field at a fixed temperature for a fixed period of time (one hour in the instant case) and cooling to a reference temperature (here room temperature of 25° C.) and then checking for magnetic property changes. This is repeated for identical time intervals but at successively higher temperatures. A careful consideration of the curves shown in the drawings disclose that regardless of whether the remanence increases slightly or decreases slightly during the irradiation step of the process, high remanence is achieved during the annealing step as shown by curves A and B. The sample of Mumetal of curve C was irradiated without the application of an external field whereas a Mumetal sample of curve D was not irradiated. Very little change in remanence occurs in the non-irradiated sample whereas the irradiated sample shows a very great increase in remanence under the same controlled conditions.

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The advantages of this low temperature-two-step process over high temperature magnetic annealing methods (thermomagnetic treatments) are (1) much lower temperatures, (2) much shorter annealing times, (3) no special furnace atmospheres are required and (4) the properties achieved in most cases are significantly better. The advantages over other irradiation treatments are that irradiation is carried out at appreciably lower temperatures, no radioactivity is induced in the materials and an externally applied magnetic field need not be present during irradiation. In addition it should be pointed out that all irradiation methods are done at remote locations; the materials are either a nuclear reactor or subjected to a beam of particles but in either case with temperature monitoring leads and coolant lines coming through heavy shielding to a remote control location. Thus control of coolant flow etc. is more difficult to achieve remotely than directly. In this invention however, irradiation is used to implant point defects in the material in the first (irradiation) step. Then the second step (post irradiation annealing treatment), is done directly in conventional furnaces but at relatively low temperatures. Thus the temperatures and times of annealing are much more amenable to control and the high values of rectangularity, remanence and maximum permeability and, in particular, the low values of coercive force can be more easily pro-

Obviously many modifications and variations of the present invention are possible in light of the above teachings

What is claimed is:

1. A method of improving the magnetic properties of a magnetically permeable material consisting of the two step process of:

irradiating the material at a temperature below 100° C. in the absence of an externally applied magnetic field to introduce a crystalline defect into the material, and

annealing said material at a temperature within the range from about 100° to 250° C. in the presence of an externally applied magnetic field until the material exhibits a substantially rectangular hysteresis loop characteristic.

2. The method of claim 1 wherein the irradiation is accomplished by irradiating the material with an electron beam.

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117—62, 227; 148—101, 102, 103, 108, 121