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MAGNETIC MATERIAL AND PROCESS OF MAKING IT

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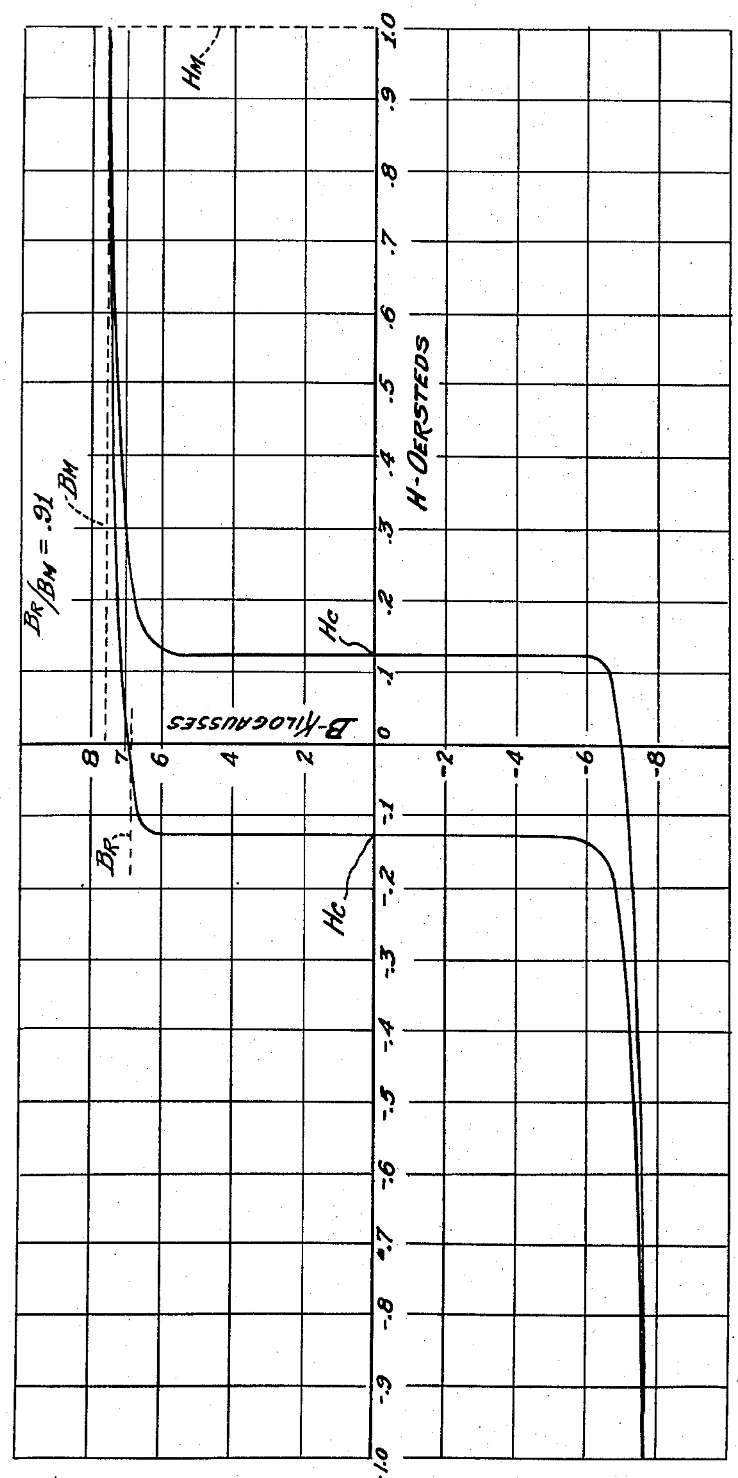

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MAGNETIC MATERIAL AND PROCESS OF MAKING IT

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9 Claims. (Cl. 148—120)

This is a continuation-in-part of my copending application, Serial No. 336,734, filed February 13, 1953, and entitled Magnetic Material and Process of Making It, to be abandoned.

My invention contemplates and has for its primary object the provision of a magnetic core material in the form of ultra-thin tape characterized by rectangular hysteresis loops. This and other objects of the invention which will be apparent to the skilled worker in the art upon reading these specifications, I accomplish by that procedure and in those alloys of which I shall hereinafter disclose exemplary embodiments.

The figure is a typical direct current hysteresis loop of my new material illustrating the degree of rectangularity achieved with magnetic tape of .25 mil thickness.

Magnetic materials having square hysteresis loops have hitherto been found of value in various fields, such as the field of mechanical rectification of alternating currents and the field of magnetic amplification. Hysteresis loop tests using direct current testing equiment are well known. A material is said to have a square hysteresis loop if, in such tests, it exhibits a high ratio of residual induction (B_r) to maximum induction (B_m). The B_r/B_m ratio for a given material is not a single value, but varies with the peak magnetizing force and passes through a maximum as saturation is approached. While the terms "square" or "rectangular" as applied to hysteresis loops may be susceptible of wide interpretation, the materials to which this invention is addressed have greater utility the higher the maximum B_r/B_m ratio becomes; and in general I contemplate materials having B_r/B_m ratio greater than .90.

High speed digital computing machines have been built using electronic vacuum tubes. While such machines are capable of solving problems of prodigious length and complexity, a serious disadvantage is to be found in the possibility of unpredictable tube failure, making rechecking impossible even though an error is known. The advent of grain oriented 48% nickel-iron alloys characterized by rectangular hysteresis loops suggested the possibility of using saturable-reactor cores to replace electronic tubes in the memory units of such computers. Successful memory units have been made using the 48% nickel-iron material having rectangular hysteresis loops in thicknesses as low as ½ mil.

Recent developments in digital computers as well as other electronic devices have necessitated higher rates of magnetization or shorter switching times within the cores than were possible with previously available magnetic tapes one mil in thickness. Since increasing the rate of magnetization is dependent upon reducing eddy currents, rolling the magnetic tape to thinner gauges would be indicated. The difficulty here is that in materials previously known, in which rectangular loops could be produced for this purpose, such as grain oriented 3% silicon-iron and grain oriented 48% nickel-iron, the coercive force increases rapidly with decreasing thickness and becomes excessive at the ultra-thin gauges required. Any increase in coercive force is undesirable, since it necessitates an increase in the exciting current, which may exceed that available.

I have made the discovery that an alloy of the 4% Mo–79% Ni type, which has a coercive force inherently lower than the other materials useful for this purpose, can be made to have a rectangular hysteresis loop at thicknesses less than one mil by proper processing, which will be described hereinafter. This was entirely unexpected as, unlike the other materials used, the 4% Mo–79% Ni alloy does not exhibit a rectangular loop in the heavier gauges. As pointed out hereinafter, some departure from these content values is permissible; but the terms "4% Mo–79% Ni" or "4–79" are convenient expressions, used herein, for indicating the general type of alloy with which I am concerned.

This discovery makes it possible to achieve much higher rates of magnetization and shorter switching times without sacrificing rectangularity of the hysteresis loop or low coercive force. This is achieved not only by reduction in gauge, but also by the higher volume resistivity and other factors not completely understood.

Therefore, a specific object of this invention is the provision of magnetic alloys of high direct current permeability at inductions approaching saturation, characterized by low coercive force, in very thin sections of high resistivity, such that eddy current effects are minimized, and characterized in such thin sections by a high B_r/B_m ratio. The most important factors involved in the provision of such material appear to be (1) composition, (2) thickness of the ultimate material, (3) the nature of the final heat treatment as respects time, temperature and annealing atmosphere, and (4) processing as respects cold rolling, intermediate anneals and the like. These factors are stated in the order of their apparent importance as respects the securing of rectangular hysteresis loops when the material is reduced to verry small thicknesses.

As to composition, the alloy from which my new material is made may contain from 75 to 85% nickel, and from 3 to 6% molybdenum, the balance being primarily iron. Manganese can be used for deoxidation and to improve hot workability and may range from around .3% to 1%, with around .6% being preferred. Silicon may also be used for deoxidation and preferably will not exceed .3% in the metal.

Sulfur should be as low as possible and preferably less than .005%, and lead less than .002%. Carbon should be low in amount, namely not more than about .1%. Aluminum should be as low as possible and preferably below .01%, including all forms present. These and other elements ordinarily present as contaminants of the iron, molybdenum, or nickel, so long as they are low in amount, do not appear to affect to any great extent the characteristics with which this application deals.

My preferred allow will contain 80%±2% nickel, and 4.0%±.3% molybdenum.

Novelty is not claimed in the constitution of the alloy since high permeability alloys within the range stated have hitherto been produced. It has not, however, hitherto been known that such a high B_r/B_m ratio could be produced in such materials.

The alloys can be formed in the electric furnace (using the induction furnace and vacuum melting if desired, although this is not always necessary). Ingots are cast and these are hot rolled to suitable intermediate gauges and prepared for cold rolling. The materials themselves are capable of being cold rolled with very heavy reductions in the absence of intervening annealings; but in the production of very thin materials it is usually easier to accomplish the cold rolling reduction in a series of stages. These stages may be widely varied.

Without desiring to limit the invention, an exemplary routing, which can be depended upon to yield my new result, includes the steps of hot rolling the material to a suitable gauge, such as 120 mils; then box annealing it at a temperature of 870° C. in a reducing atmosphere, such as dry hydrogen or dissociated ammonia; cold rolling it to a thickness of 25 mils; open or strand annealing it in a reducing atmosphere at 870° C.; cold rolling it to a thickness of 1.2 mils; again open annealing it at 870° C. in a reducing atmosphere; and cold rolling it to the final gauge of .25 mil. It is then ready for a final anneal.

An exemplary routing to produce ⅛ mil material is the same except that the intermediate anneal is practiced at a thickness of .5 mil, after which the strip is rolled directly to .125 mil before final annealing.

These are exemplary routings only, intended for the manufacture of ¼ mil and ⅛ mil materials. They can be widely varied even for the production of material of the same final thickness, such as by the omission of the last intermediate anneal. Obviously, it may be varied for the manufacture of stock of other thicknesses, say ½ mil or lighter than ⅛ mil. The particular sequence of rolling stages and intermediate anneals has been found to be of only secondary importance in producing rectangularity; and this is true also of the number of cold rolling stages by which the material is reduced to final gauge. The reducing annealing atmospheres are to avoid oxidation and pickling.

From the standpoint of securing rectangular hysteresis loops in thin materials of the compositions set forth, there is some advantage in subjecting the material to an intermediate anneal at a gauge which leaves a 70 to 85% reduction still to be accomplished in the final stage of rolling. The use of intermediate anneals also aids materially in softening the material for easier rolling.

The rolling of the materials, at least after they have attained light gauges will, of course, be carried on on suitable precision equipment, which means a rolling mill having very small work rolls and in which deflection of the mill elements transversely of the work piece is minimized. Excellent results can be attained in relatively narrow widths on mills of the four-high type where the very small work rolls are backed by backing rolls of large diameter and where means are provided to prevent deflection of the work rolls in the direction of movement of the strip. Where the material is to be handled in wider widths, I prefer to use a mill of the beam backed type such as that shown in Sendzimir Patent No. 2,170,732.

The material treated as described is now in condition to be given its ultimate magnetic characteristics by a final anneal such as that hereinafter described. The material is itself very delicate and difficult to handle. While it may be treated in bulk to give it the ultimate magnetic characteristics, this ordinarily is not desirable because subsequent operations of handling, slitting, winding into cores and the like will very greatly impair these characteristics. As a consequence, the formation of complete cores with the materials of this invention normally is practiced prior to the final anneal. For this reason and also because the cores themselves are ordinarily quite small and may be made in very large numerical quantities especially for such uses as the memory sections of digital computing machines, the fabrication of the material and the cores and the final anneal will most usually be practiced not by the manufacturer and roller of the material itself, but by his vendee. Thus the material, treated as hereinabove described, and coiled or otherwise handled in bulk or in sheets, is a commercial article of manufacture even though its primary utility lies in the fact that when it is given a suitable heat treatment, it will develop or possess the characteristics herein set forth as desirable.

As indicated, the cores ordinarily made are quite small, those intended for digital computing machine work being frequently no greater in diameter than about ¼ in. In my current practice the alloys are rolled to gauge as single strands in a width of about 2 in. employing work rolls of very small diameter. In the formation of the particular exemplary coils, the material, after rolling, is slit into ribbons ⅛ in. to ¼ in. in width for widening into toroidal cores. Because of the fragile nature of the material itself and of these small cores, which often consist of only a few wraps of the ribbon, it was found advantageous to wind the cores on tiny ceramic bobbins such as bobbins of steatite. These bobbins, which have lateral flanges, furnish support for the ribbon during annealing and also provide a frame for the copper windings employed in testing and in many instances in the subsequent use of the cores. The convolutions of the metallic ribbon are carefully insulated with a magnesia coating prior to annealing.

The annealing is conducted in a suitable annealing box in an atmosphere predominantly of hydrogen and substantially free of carburizing, sulfurizing and nitriding impurities. The water vapor should be as low as possible and such that the exit dewpoint will lie below −40° C. and preferably below −50° C. The annealing temperature may range from 750° C. to 1050° C., my preference being for temperatures around 850 to 975° C. when the soaking time is two hours. While my preference is for pure hydrogen, other inert gases may be present in appreciable quantities. For example, I may employ dissociated ammonia. The nitrogen present is dissociated ammonia does no harm; but any substantial quantity of undissociated ammonia should be avoided because of its nitriding effect.

The preferred annealing temperature represents the optimum balance between maintaining the rectangularity of the hysteresis loop and minimizing the coercive force, in order to obtain the shortest possible switching time with adequate signal-to-noice ratio under pulse excitation. This balance is necessary because while higher temperatures reduce the coercive force they also reduce the rectangularity.

I have found that it is not necessary to hold the material at the annealing temperature except for a sufficient time to insure that all parts of the charge have come to a uniform temperature. Generally one hour is sufficient for the small cores of this material usually being annealed. A relatively brief soak at full temperature may be tolerated, e. g. one to two hours, but a more prolonged soak should be avoided.

The material may be furnace cooled, which results in a slow cooling throughout the range substantially to room temperature, or it may be quenched from a temperature not substantially lower than 540° C., provided the cooling is carried out in such a manner as to avoid oxidation harmful to magnetic properties, as for example by maintaining the material in an atmosphere of hydrogen.

The annealing temperature range, as well as the moisture content of the annealing atmosphere, are critical, and for best results should not be departed from substantially.

It has been found that this treatment results in a material having excellent magnetic characteristics and a very high B_r/B_m ratio and low coercive force in spite of the extreme thinness of the material.

This behavior of the particular alloys of the invention is curious and unexpected. In 3% silicon-iron the coercive force increases substantially in proportion to the reduction in thickness. Reducing the thickness of 48% nickel-iron also increases the coercive force but not as rapidly as is the case with silicon-iron. Reducing the thickness of the 4% Mo–79% Ni alloy not only increases the coercive force appreciably less than in these other materials, but this increase is no longer a limiting factor, as the coercive force of this alloy is so much lower at any gauge. Further, while both with the 3% silicon-iron and the 48% nickel-iron alloys the hysteresis loops can be made quite rectangular at one mil thickness and above, it is not until the thickness is less than one mil that the 4–79 material can be made to exhibit loops of good rectangularity. Hence the high degree of rectangularity of the hysteresis loops which I have obtained in this material at ultra-thin gauges could not have been predicted in advance.

The coercive force in my preferred materials at .25 mil thickness is about .10 oersted when the tip magnetizing force H_m is .25 oersted.

Direct current tests performed on an exemplary material of this invention gave values as follows:

H_m Oersteds	B_m Kilogausses	B_r Kilogausses	B_r/B_m	H_c Oersted
.25	7.35	6.8	.93	.093
1	7.85	6.9	.88	.093

As has been indicated above, the alloy compositions to which my invention is addressed contain the specified percentages of nickel and molybdenum, the balance being substantially all iron, which may contain normal amounts of impurities or minor amounts of other alloying ingredients whether occurring as impurities or added for workability. It has been further pointed out that these minor amounts of other alloying ingredients do not materially or substantially affect the magnetic characteristics of the materials as discussed herein. As a consequence, in the claims which follow, the expression "balance substantially all iron" has been employed to be inclusive of such minor amounts of these other alloying ingredients as may be present within the scope of my disclosure.

Modifications may be made in my invention without departing from the spirit of it. Having thus described my invention in certain exemplary embodiments, what I claim as new and desire to secure by Letters Patent is:

1. A process of producing exceedingly thin magnetic alloys having high permeability, low coercive force and rectangular hysteresis loop characteristics, which comprises reducing an alloy containing 80±2% nickel and 4.0±.3% molybdenum, balance substantially all iron, to a thickness less than 1 mil and subjecting the reduced material to an anneal in an atmosphere predominantly of hydrogen and having a dew point below −40° C. at a temperature of substantially 750° to 1050° C.

2. A process of producing exceedingly thin magnetic alloys having high permeability, low coercive force and rectangular hysteresis loop characteristics, which comprises reducing an alloy containing 75 to 85% nickel and 3 to 6% molybdenum, balance substantially all iron, to a thickness less than 1 mil and subjecting the reduced material to an anneal in an atmosphere predominantly of hydrogen and having a dew point below −40° C. at a temperature of substantially 750° to 1050° C.

3. The process claimed in claim 1 wherein the material is reduced to final gauge from an intermediate hot rolled gauge in a series of stages with intermediate annealings, the last of said stages occurring at a gauge requiring about a 70 to 85% reduction to attain final gauge.

4. The process claimed in claim 1 wherein the final gauge of said material is substantially ¼ mil.

5. The process claimed in claim 1 wherein the final gauge of said material is substantially ⅛ mil.

6. The process claimed in claim 2 wherein the material is wound into a toroidal coil in the presence of magnesia as an anneal separator and is supported in toroidal form by an internal heat-resistant support prior to the said final heat treatment.

7. A process of producing a magnetic material of high permeability, low coercive force and of a thickness minimizing eddy current losses at high frequency magnetization, which comprises subjecting an alloy consisting of 80±2% nickel, 4.0±.3% molybdenum, balance substantially all iron, to cold rolling from an intermediate gauge to reduce it to a thickness substantially less than 1 mil with intermediate open annealings, and subjecting the material to a final heat treatment in an atmosphere predominantly of hydrogen at about 850 to 975° C. so as to develop a maximum B_r/B_m ratio of at least about .90 when tested with direct current at a magnetization parallel to the direction of rolling.

8. The product produced in accordance with the process of claim 1.

9. The product produced in accordance with the process of claim 2.

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