March 22, 1938. A. A. FREY ET AL 2,112,084

MAGNETIC MATERIAL AND METHOD OF PRODUCING THE SAME

Filed Nov. 1, 1934 2 Sheets-Sheet 1

Fig. 1.

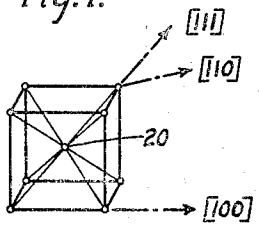


Fig. 2.

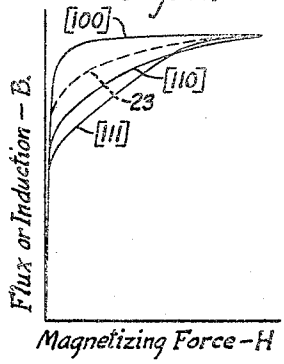


Fig. 3.

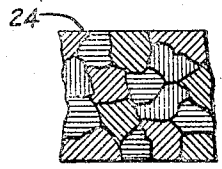


Fig. 4.

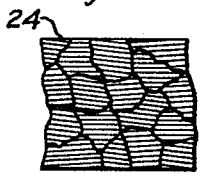


Fig. 5.

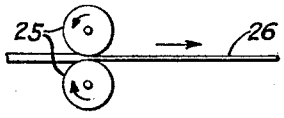


Fig. 6.

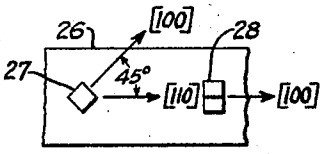


Fig. 7.

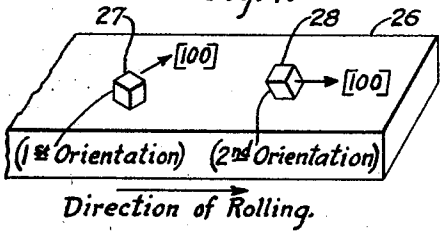


Fig. 8.

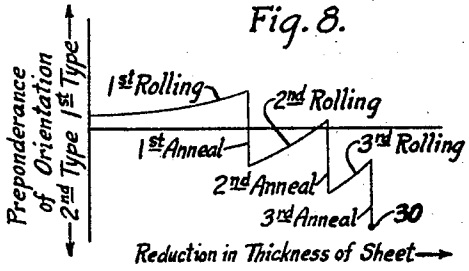
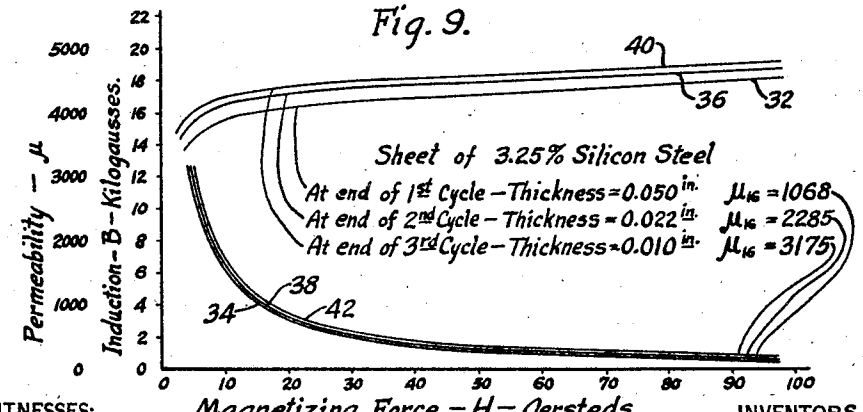


Fig. 9.



WITNESSES:

Leon J. Vaza

L. T. Bryant

INVENTORS.

Albert A. Frey and Francis Bitter.

BY

Ezra W. Farago

ATTORNEY

March 22, 1938. A. A. FREY ET AL 2,112,084

MAGNETIC MATERIAL AND METHOD OF PRODUCING THE SAME

Filed Nov. 1, 1934 2 Sheets-Sheet 2

Fig. 10.

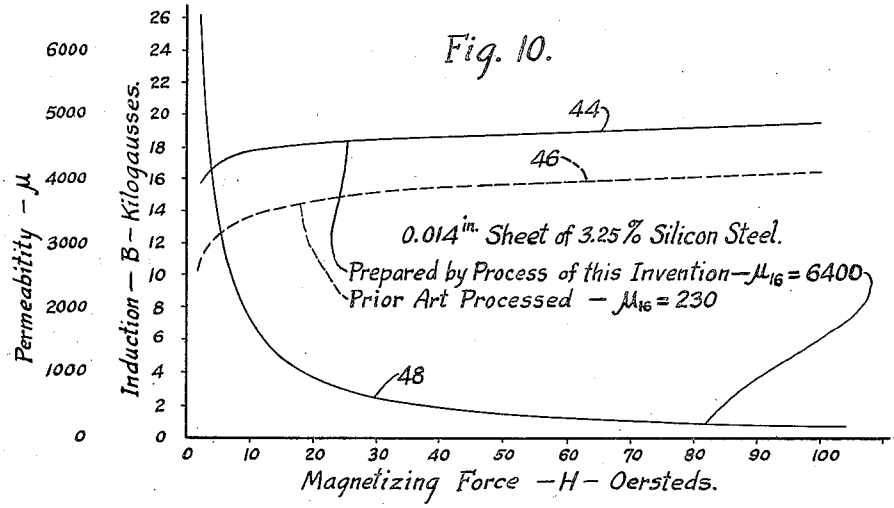


Fig. 11.

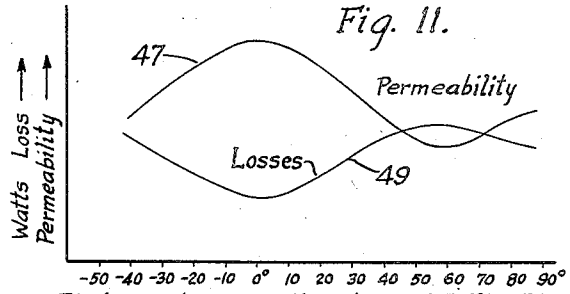


Fig. 12.

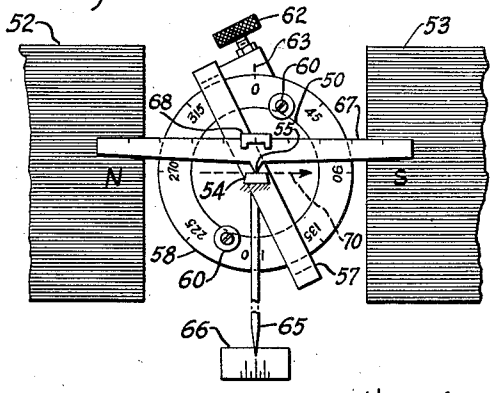


Fig. 13.

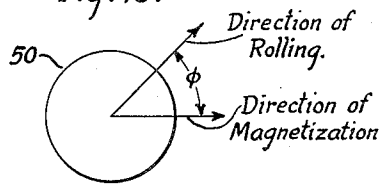
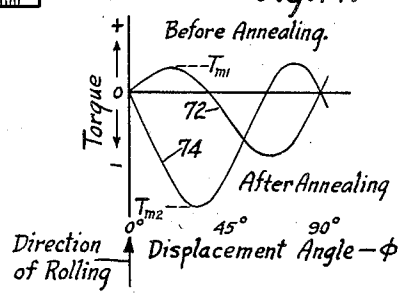


Fig. 14.



WITNESSES:
Leon J. Zaza
L. F. Bryant

INVENTORS.
Albert A. Frey and
Francis Bitter.
BY
Ezra W. Savage
ATTORNEY

Patented Mar. 22, 1938 2,112,084

UNITED STATES PATENT OFFICE

2,112,084

MAGNETIC MATERIAL AND METHOD OF PRODUCING THE SAME

Albert A. Frey, Wilkinsburg, Pa., and Francis Bitter, Cambridge, Mass., assignors to Westinghouse Electric & Manufacturing Company, East Pittsburgh, Pa., a corporation of Pennsylvania

Application November 1, 1934, Serial No. 750,930

7 Claims. (Cl. 148—12)

This invention relates to magnetic materials and it has particular relation to an improved character of and one preferred process for producing ferro-magnetic materials in sheet-like form which exhibit enhanced permeability and other magnetic properties at high flux densities.

Magnetic material in laminated or sheet form is extensively utilized in the manufacture of transformers, induction regulators, dynamo-electric machines, and other electrical apparatus in which substantial quantities of magnetic flux must be linked with electrical conductors. The electrical steel of commerce today is an alloy of low carbon steel and silicon, in which the silicon content ranges from about ½% to about 5%, which alloy is processed into sheets of a thickness of the general order of 0.014". The upper limit in the practical thickness range for the sheets depends upon the eddy-current loss which is permissible in the magnetic circuits to be built up therefrom, while the lower limit is dictated by the cost with which the sheets may be made and handled.

To reduce the size, weight and cost of the referred-to apparatus, the physical dimensions of the flux-conducting or magnetic circuit parts should be kept as low as possible. Hence it is preferable that magnetic material for use in such circuits possess high permeabilities at high flux densities, since in building transformer cores for example, the higher the high density permeability of the material employed the smaller the transformer may be made for a given capacity. In the interests of apparatus efficiency, the ideal material should also have relatively low hysteresis and other power loss characteristics.

Generally stated, the object of this invention is to produce ferro-magnetic materials in sheet form which are of greatly increased utility in the manufacture of electrical apparatus.

A more specific object of this invention is to provide an improved method for processing sheets of ferro-magnetic material to produce in them high permeabilities at high flux densities together with relatively low hysteresis and other power losses.

This invention is predicated upon the discovery that the sheet or other form of magnetic material having the highest permeability at high flux densities is that in which the grains are so oriented that the direction of easiest magnetization through the grain crystals coincides with the direction of magnetization of the material sheet. This invention specifically contemplates the production of an especially advantageous form of grain orientation in sheets of ferro-magnetic material, which orientation is attained by subjecting the sheet during its final stages of thickness reduction to one or more specially predetermined cycles of strain introduction and annealing which combine to produce a well-fibered material having large grains due to recrystallization.

While preferably these strains are of the permanent elastic type produced by a plastic deformation of the material they may instead be those which result from a proper mechanical stressing, as a combined compressing and stretching action, of the material during the anneal. Those of the first-named variety may conveniently be introduced into the material by a conventional rolling operation carried on at a temperature below that of recrystallization for the given material. In the case of iron-silicon alloys this rolling should, to be most effective, preferably effect a reduction in sheet thickness of from about 30% to 60%, and may satisfactorily be performed at room temperature. For the named material, the annealing operation is preferably carried on at a temperature of the general order of from about 700° C. to 1300° C. for a period which, depending upon the nature of the material and certain other factors, may vary from a minimum of several seconds to a maximum of several hours. The rolling so strains the material crystals that the subsequent annealing in relieving those strains orients the crystals with the lateral edges of the cubic structures substantially parallel to the direction of rolling. The result is sheet material which exhibits both the highest permeability and lowest loss characteristics in the direction of rolling.

Not only is this strain-annealing treatment useful in producing in material originally isotropic an advantageous type of fibering and grain orientation but it may also be applied to retain or stabilize fibering originally in the material as a result of other treatment, of which a cross-rolling process, more completely disclosed and described in a copending application, Serial No. 744,816, filed September 16, 1934, by F. Bitter, may be cited as an example. In that process, the material is reduced to its final thickness by rolling in two different directions which are displaced from each other by substantially 90°, and the best magnetic properties are in a direction displaced 45° from these directions of rolling. By further rolling such fibered material in the 45° direction the original orientation may be largely preserved during the final high temperature heat

treatment which is necessary to develop permeability and reduce hysteresis losses.

When manufactured by ordinary sheet-mill practice, the permeability of laminated silicon steels of the grades commonly utilized in the building of electrical apparatus decreases to relatively low values at the higher densities at which it is found advantageous to operate such equipment, it being of the order of 250 at a flux density of 16,000 gausses. Such material, which may be characterized by large or small grain size depending upon the particular process adopted by the manufacturer, has been made with a guaranteed 60-cycle watt loss of 0.55 to 0.60 watt per pound at 10,000 gausses for 14 mil sheets. When commercial steel comparable to that above mentioned is produced in accordance with the improved process of the present invention, a permeability of as high as 6,400 is observed at a flux density of 16,000 gausses. The 60-cycle watt loss at 10,000 gausses maximum flux density is as low as 0.33 watt per pound for 14 mil sheets of the same material. Since this material may be produced through the application of easily carried out rolling and annealing operations, and since the enhanced characteristics are best in the direction of rolling, it will be appreciated that the process of this invention is one of an exceedingly practical and otherwise valuable nature.

The invention itself, together with additional objects and advantages thereof, will best be understood through the following description of a specific embodiment when taken in conjunction with the accompanying drawings, in which:

Figure 1 is a representation of the manner in which the atoms are arranged in a single crystal of magnetic material of the class typified by iron;

Fig. 2 is a diagram of curves illustrating certain magnetic characteristics of the crystal of Fig. 1;

Figs. 3 and 4 are representations of magnetic materials respectively having a random and a preferred orientation of the axes of their grain or crystal structures;

Fig. 5 is a simplified representation of the conventional manner in which a sheet of magnetic material is passed between two pressure rolls for the purpose of reducing its thickness;

Fig. 6 is a plan view of the sheet of Fig. 5 upon which is indicated two different manners in which the individual crystals in the material appear to orient themselves during the treating cycle of the present invention;

Fig. 7 is a perspective view of an enlarged portion of the sheet of Fig. 6, in which the two types of crystal orientation are further illustrated;

Fig. 8 is a diagram of curves showing the manner in which the degree of the two types of orientation appears to progressively change when the material is subjected to a plurality of cycles of strain annealing;

Fig. 9 is a diagram of curves illustrating the progressive enhancements in permeability produced in a sheet of 3.25% silicon steel when treated by the three cycles of cold rolling and annealing represented in Fig. 8;

Fig. 10 is a diagram of curves illustrating the magnitude of improvement in characteristics exhibited by a sheet of 3.25% silicon steel when processed in accordance with the present invention under optimum conditions to a final thickness of 0.014 inch;

Fig. 11 is a diagram of curves illustrating the high preference to the direction of rolling which the magnetic characteristics of material produced by the process of this invention exhibit;

Fig. 12 is a simplified representation of one form of magnetic testing apparatus which may be utilized to indicate the degree of fibering and type of crystal orientation in sheet magnetic material;

Fig. 13 is a diagram showing how the position of a circular specimen of the material is varied during test by the equipment of Fig. 12; and

Fig. 14 is a diagram of curves depicting the comparative results of tests made by the apparatus of Fig. 12 upon magnetic steel before and after the annealing operation in one of the treating cycles of the process of this invention.

Referring to the drawings, the relative positioning of the atoms in a single crystal of magnetic material of the class typified by iron is there illustrated in Fig. 1. In such material, as well as in that which is typified by nickel, the crystal is in the form of a cube having an atom positioned at each of the eight corners thereof. In the iron class of materials, which include silicon steels and many of the other commercially known magnetic alloys of iron, each crystal, which is commonly termed a body-centered cubic structure, in addition has at the geometric center thereof another atom indicated in Fig. 1 at 20. In the class of materials typified by nickel and combinations of nickel and another metal, such as iron, up to certain given or critical percentages, each crystal, which is commonly termed a face-centered cubic structure, has an additional atom at the center of each of the six faces thereof.

It is known that such cubic crystals possess different magnetic characteristics along different of their axes, which differences are for the structure of Fig. 1 when in an unstrained condition represented by the magnetization curves of Fig. 2. Considering this structure, the direction of easiest magnetization, to which the curve [100] of Fig. 2 applies, is along any one of the three tetragonal axes, one of which is indicated in Fig. 1 at [100] and each of which defines a direction perpendicular to a face of the structure. Ranking next in ease of magnetization is any one of the directions from one corner of a face to a diagonally opposite corner of the same face. The diagonal axis of one such direction is indicated in Fig. 1 at [110], which symbol also designates in Fig. 2 the applicable magnetization curve. Ranking last in order of magnetizability as typified by curve [111] of Fig. 2 is any one of the directions diagonally through the cube determined by any one of the trigonal axes, one of which is indicated in Fig. 1 at [111], drawn from one corner of the structure to the diagonally opposite corner.

The characteristics of Fig. 2 are exhibited not only by pure iron, but also by alloys of iron with small amounts of any element that will go into solution therewith. Examples of such elements are silicon, which, as already indicated, at the present time finds the greatest commercial application in ferro-magnetic materials, and also aluminum, nickel, copper, manganese and others. The face-centered or nickel class of crystal structure differs in that its direction of easiest magnetization is determined by the [111] axis and its direction of most difficult magnetization by the [100] axis. Such a reversed relation exists for nickel in the pure state and for alloys of nickel and iron up to approximately 20% iron, at which point the [100] and [111] curves sub-

stantially coincide. Increase in the percentage of iron above 20% in a nickel base alloy causes the crystal characteristics to start to approach those depicted in Fig. 2 for the body centered structure.

Magnetic materials known to the prior art have been of the non-fibered or randomly oriented grain variety, and when subjected to examination as by an X-ray, elastic deformation, magnetic or optical testing apparatus in manners to be further explained, reveal the condition which is represented in Fig. 3. In that figure, each of the cross-hatched areas 24 represents an individual grain of the metal and the parallel lines drawn through each of these areas are assumed to indicate the direction of easiest magnetization through the grain. Each of these grains is made up of a large number of the cubic crystal structures previously described which are arranged side by side in a regular or parallel manner. In effect, therefore, each grain assemblage exhibits magnetic characteristics comparable to those of the individual crystal structures of which it is made up. Consequently, each grain 24 is possessed of the three different types of axis [100], [110] and [111] in the respective directions of which the magnetizing characteristics are for body centered crystal material as depicted by the curves of Fig. 2.

For the mentioned random orientation of the crystal grains, distribution of the grain axes is substantially the same in all directions and no particular direction can, therefore, be said to be preferred. Consequently, the highest obtainable magnetic property of material of the non-fibered or randomly oriented grain variety of Fig. 3 may be expected to be of some average order, such as is indicated by the dotted curve 28 in Fig. 2, which, in effect, represents an average of the three full-line curves. The fact that such a random orientation, or a substantial approach thereto, has been characteristic of prior art magnetic materials explains the inferior magnetic characteristics of such materials as compared with those prepared in accordance with the teachings of the present invention.

When, however, certain of the axes of the individual crystal grains 24 are all lined up in substantially the same direction, as is indicated by the showing of Fig. 4 which typifies a condition spoken of as fibering, the material is found to exhibit preferential directions of easy magnetization, which directions are taken advantage of in the practice of the present invention. In achieving the objects of this invention, the desired orientation of the material grains is, as before pointed out, affected by subjecting the material to one or more specially predetermined cycles of rolling and annealing. This orientation, which will be further explained by reference to the representations of Figs. 5 to 7, inclusive, is one in which the lateral edges of the cubic crystal structures appear to coincide with the direction of sheet rolling while the plane surfaces of the cubes which these edges define appear to be randomly oriented about the axis of this rolling direction.

The rolling operations may in the process of the present invention be performed in the manner represented in Fig. 5 in which a sheet of material 26 is shown as being passed between a pair of suitably driven compressing rolls 25. It is observed that such a passage functions to orient, in the case of a ferro-magnetic material, the [110] axes of the crystal structures, one of which, in greatly magnified form, is represented at 27 in Fig. 6, in a direction parallel to the direction of rolling. As a consequence, certain of the [100] axes of the crystals tend to line themselves up, as is also indicated in Fig. 6, in a direction displaced by 45° from the direction of rolling, and it is thus observed that in a sheet of ferro-magnetic material rolled in a single direction the direction of easiest magnetization is similarly displaced. In the case of silicon steel of the now commercially utilized grades a reduction in sheet thickness of from about 30% to 60% by such rolling has been found to be the most effective in introducing the character of strains which are of the greatest advantage in achieving the objects of the present invention. In all cases reductions substantially greater than the small values, of one or two percent, for example, sufficient to effect recrystallization merely are found essential.

In effecting this reduction the strip may be passed through the rolling mill a number of times and the amount by which the thickness is reduced by each pass may be varied as is most convenient. In some cases, a slight preheating, as in a bath of oil to a temperature of from 150° to 200° C., may be of advantage in rendering the iron more malleable to facilitate this rolling.

It is desired to here point out that when materials of the nickel or face-center crystal class are similarly rolled, a somewhat different result is produced, the tendency being for the crystal structures to line up with their [111] axes parallel to the direction of rolling. For this class of materials, the phenomenon is considerably more complicated than is that of the body-centered cubic crystal material about which much more at the present time is known and with which the present invention is most directly, although not exclusively, concerned.

The heat-treating or annealing which follows the described rolling of the material may in the process of the present invention be effected by placing the material in a furnace in which the temperature ranges from about 700° to 1800° C. for a period of time which for some conditions may be very short and for others of the order of several hours. A presence in the annealing furnace of hydrogen or other reducing atmosphere is found to be of advantage.

Assuming that the rolled sheet 26 of Figs. 5 to 7 is of silicon steel or other ferro-magnetic material, such an annealing treatment is found both to relieve the previously set up internal strains and to re-orient or shift a substantial portion of the crystals into the position indicated at 29 in Figs. 6 and 7. This second type of orientation, in which certain of the [100] crystal axes coincide with the direction of rolling, is that which is depended upon in producing the materially enhanced characteristics by the process of the present invention. Its presence in material prepared by this improved process is evidenced by X-ray, magnetic, elastic and optical tests methods, all of which, depending upon quite different things, check each other, as will be made more evident presently. By such means, it may be demonstrated that, while two of the six faces of the crystal structure 29 are uniformly aligned in the planes perpendicular to the sheet rolling, the remaining four of the structure faces are not so uniformly arranged, their positioning apparently being of a random nature. The existence of such a relation is further substantiated by the fact that materials prepared by the proc-

ess of the present invention exhibit their best magnetic characteristics in the direction of rolling and show considerably inferior properties in all other directions. The magnitude of this directional preference is generally indicated by the curves of Fig. 11 to be further discussed.

A proper choice of the initial and final thickness of the sheet to be treated and the conditions under which the rolling and subsequent annealing operations are administered is found to effect very favorable enhancements when only one cycle of rolling and annealing is applied to the material. In many cases, however, it is observed that the greatest improvement may be effected when more than one cycle is applied, and in Fig. 8 there is illustrated the changes in the two types of crystal orientation which appear to result when three cycles of the treatment of the present invention are applied to a material. In Fig. 9 the corresponding changes in magnetic characteristics in a sheet of 3.25% silicon steel reduced during these three cycles from an initial thickness of 0.075" to 0.010" are likewise shown.

During the first rolling operation of the treatment program of Fig. 8, the type of orientation typified by the crystal **27** of Figs. 6 and 7 is introduced into the sheet and practically none of the second type typified by crystal **28** appears to result. When subsequently annealed, however, as the strains in the material disappear, most of the first type of orientation is found to disappear and a considerable preponderance of the second type is evidenced. Further reduction of the sheet by a second rolling then tends to restore the first type of orientation, which restoration is destroyed or converted into the second type of orientation by a second annealing process, at the end of which the preponderance of this second variety of crystal positioning is considerably greater than at the end of the first annealing period. A third rolling operation in turn likewise tends to restore the orientation to the first type, while a third or final annealing again increases the preponderance of the second type of orientation, bringing it to a final point **30** which represents a greater degree of completeness than at the end of the first or second anneals, as the curves of Fig. 8 generally indicate.

Considering the application of the treating program of Fig. 8 to the 3.25% silicon steel whose characteristics are depicted in Fig. 9, in the original preparation of this material stock of a commercial grade of 3.25% silicon iron alloy was first melted in a suitable furnace and then allowed to solidify into an ingot. This ingot was reduced by standard and previously known processes of forging and rough rolling at temperatures of the general order of 1000° C. into a plate-like sheet bar. This bar was next hot rolled at a temperature of between 800° C. and 1000° C. into match plates. By hot rolling, these plates were further reduced into strips 0.075" in thickness to which strips the program of strain-annealing referred to was applied.

The first step in this application consisted in rolling at room temperature these 0.075" sheets to a thickness of 0.050". It may here be pointed out that the critical or most effective percentage of reduction depends upon the amount of alloying constituent, the nature thereof, and the degree of impurity in the alloy.

The 0.050" sheet thus rolled, was then annealed, at a temperature of the general order of 1200° C. for a period of time sufficient to recrystallize the small grains into considerably larger ones. At the end of this first anneal, the B—H or magnetization curve was as depicted at **32** in Fig. 9, while the corresponding permeability curve was as depicted at **34** in the same figure. The curve last named was derived from the one first named by applying the equation:

$$\text{Permeability } (\mu) = \frac{\text{Induction (B) in gausses}}{\text{Magnetizing force H in oersteds}}$$

At a representative density of 16,000 gausses, the material thus treated exhibited a permeability of 1068.

To further improve its characteristics, this 0.050" sheet was then subjected to a second cold rolling operation, which reduced its thickness to 0.022", a reduction of slightly more than 50%. A second annealing was then effected at a temperature slightly lower than that of the first or of the general order of 1000° C., such reduction being found advisable to prevent excessive grain growth. At the end of this second annealing, the material exhibited the magnetization characteristics depicted by the curve **36** of Fig. 9 and corresponding permeability characteristics depicted by the curve **38**. At the previously chosen representative flux density of 16,000 gausses, the permeability was, by the second cycle of rolling and annealing, raised to 2285.

A third cycle of rolling and annealing was then applied during which the material was reduced from a thickness of 0.022" to 0.010", and at the end of this cycle, the material exhibited the magnetization and permeability characteristics depicted by curves **40** and **42**, respectively. As a result of this third cycle of treatment, the permeability at the representative density of 16,000 gausses was raised to 3175. In the pictorial representation of the three cycles of treatment of Fig. 8, this corresponds to the final condition indicated by point **30**. The final 0.010" sheet thus prepared was found to exhibit relatively low power loss characteristics, the magnitude of which was considerably less than the same material when processed by prior-art methods.

In a multi-cycle program of treatment of the character just detailed, the temperature and duration of the intermediate annealing operations are preferably so selected as to produce a material of relatively fine grained nature. The final annealing treatment, however, is found to be most effective in enhancing magnetic characteristics when carried on at a relatively high temperature, of the general order of 1200° C. for a relatively long period, of the general order of several hours.

When 3.25% silicon steel is reduced to a final thickness of 0.014" and treated under optimum conditions in accordance with the teachings of the present invention, magnetization characteristics depicted by curve **44** of Fig. 10 have been observed. As compared with curve **46**, which applies to the same material when processed by prior art methods, the substantial improvement made possible by this invention is at once evident. Whereas such prior art processed material exhibits at 16,000 lines per square centimeter a permeability of about 230, and has a watt loss at a 60-cycle flux of maximum density of 10,000 gausses of about 0.45 watt per pound, the same material when properly prepared in accordance with the present invention has, as reference to the permeability curve 48 which applies thereto will show, a permeability at 16,000 gausses of approximately 6400. Although not shown by curve **48**, at zero flux this improved material ex-

hibits a permeability of 2000, and a maximum permeability of approximately 60,000. At a 60-cycle flux having a maximum intensity of 10,000 gausses the watt loss is only 0.33 watt per pound.

In preparing the material whose characteristics are depicted by curves 44 and 48 in Fig. 10, the procedure was as follows: An ingot was prepared by melting stock of 3.25% silicon steel in hydrogen, the presence of which prevented excessive oxidation and removed appreciable quantities of carbon and sulphur. This ingot was forged into billets which were hot rolled in a commercial strip mill to a thickness of 0.125". The temperature during the final passes of this rolling was maintained between 650° and 900° C. This 0.125" strip was then pickled by immersing in sulphuric acid for a time sufficient to thoroughly clean the surface thereof.

During the first stage of the strain-annealing treatment the 0.125" strip was reduced to 0.061" by rolling at a temperature of approximately 150° C. It was then annealed in a furnace containing hydrogen the temperature of which was raised to between 850° and 900° C., held at that point for about 15 minutes, and then allowed to cool to room temperature. In the particular furnace used, approximately two hours were required to bring the temperature up to and down from the stated values.

A second stage of treatment was then applied during which the 0.061" strip was, under the above stated conditions, further cold rolled to approximately 0.030" in thickness, and then reannealed in a manner also the same as that set forth above. This was followed by a third and final stage of treatment during which cold rolling to a thickness of 0.014" was effected and an annealing in hydrogen at a temperature of 1200° C. for approximately 10 hours was given. The 0.014" sheet thus prepared exhibited the characteristics of Fig. 10.

From the test results just presented, the marked improvement in the high density permeability and power loss characteristics which ferro-magnetic material sheets prepared in accordance with the present invention exhibit is at once apparent. For electrical uses, as already indicated, improvement within these high flux density ranges is of particular practical importance and advantage. Furthermore, a processing of the raw material in accordance with the teachings of this invention is simple and inexpensive and can be accomplished rapidly and efficiently.

Nor are the beneficial results restricted to the 3.25% silicon steel specifically mentioned, since when applied to other comparable materials containing different proportions of silicon equally enhanced characterstics have been observed. The distribution of cold working and heat treatment, however, must be varied for different percentages of silicon and in accordance with the type and characteristics of the particular hot worked material being used. The gauge of the strip when subjected to the cold rolling and annealing process of the present invention may be of almost any commercial magnitude, though preferably not greatly in excess of 0.125" in thickness. This thickness, of course, will depend upon the manner in which further reduction to the final gauge is to be carried out, an important factor in which manner is the number of cycles of treatment which it is desired to apply.

In the description thus far given it has been assumed that the material was non-fibered or isotropic at the beginning of application of the strain-annealing process which process thus directly produced the advantageous final form of grain orientation. In addition, however, the treating program of this invention may be utilized to stabilize fibering originally in the material as a result of other treatment. Considering as an example of such earlier treatment the before referred to cross-rolling process which produces the best magnetic properties in a direction displaced by 45° from the two 90° displaced rolling directions, a final heat treatment is there necessary to develop the magnetic properties by relieving the internal strains. While a low temperature anneal (at about 800° C.) gives the highest permeability characteristics it allows objectionably high hysteresis and other power losses to persist. While a higher temperature treatment (at about 1000°) lowers the losses, it objectionably decreases the permeability.

It has been discovered that by further rolling, as in accordance with this invention, the cross-rolled material in the 45° direction the original orientation may be considerably stabilized and the final heat treatment may without appreciably destroying this orientation be carried on at a temperature sufficiently elevated to effectively reduce the material losses. As a result the advantages of both the elevated permeability and lowered losses may be availed of in the cross-rolled material.

In this application even small reductions in sheet thickness have been observed to be of appreciable effectiveness. For example a 10% reduction by cold rolling in the 45° direction caused after a 1200° C. anneal the material to increase its permeability at a flux intensity of 15,000 gausses from about 500 to in excess of 850. When reduced in thickness by a similar amount by processing in one of the two directions of original cross-rolling, the permeability after the 1200° C. anneal was in that direction increased from about 250 to in excess of 400 at the named density.

Larger reductions in thickness are observed to effect beneficial results of correspondingly greater magnitude. Hence, by choosing a proper cycle for cross-rolled material cut in a 45° direction, it is possible to rather completely stabilize the grain orientation against destruction during a final high temperature heat-treatment.

There are a number of evidences of the earlier-referred to special or second type of orientation from which the noted improvements in magnetic characteristics definitely appear to result. Important among these evidences is the directional preference in its magnetic characteristics which the treated sheet material exhibits. This is illustrated in Fig. 11, in which curve 67 shows the manner in which the permeability of the material decreases as the direction of magnetization is displaced from the rolling direction. Curve 69 likewise indicates the marked increase in loss characteristics of the material under the same conditions. Both curves apply in a general way throughout the entire range of magnetizing force or flux densities, and for this reason no particular scale has been assigned to the vertical axes thereof.

This special orientation of the material grains also evidences itself in a number of other manners, certain of which may be observed by the use of X-ray, optical, elastic deflection and magnetic testing apparatus. All of these methods, while depending upon quite different properties of the

material, are as before indicated found to very closely check each other in establishing the presence of the type of orientation under course of discussion. Description of only one of these methods, the magnetic testing one, will therefore here suffice. An equipment for the making and representative results of this magnetic type of test are illustrated by Figs. 12 to 14, inclusive.

The preferred form of equipment shown in Fig. 12 requires that the specimen of material to be tested be in the form of a circular disc indicated at **50**. This equipment comprises a magnetic circuit which includes a pair of separated pole pieces **52** and **53**, between which a very high intensity of uni-directional magnetic flux is caused to flow by suitable exciting means (not shown). Supported from a pair of stationary supports, the front one of which is shown at **54**, by cooperating knife edges **55**, is a yoke structure **57**, which carries a member **58** into a suitably formed depression in which the circular specimen **50** of the material to be tested may be fitted and secured by means of screw **60**. In the secured position, the sample lies within a vertical plane which coincides with the direction of flux passage between the two pole pieces.

By means of an adjusting screw **62**, the sample carrying member **58** may be rotated relative to the supporting yoke **57**, the amount of such rotation being indicated in degrees by the scale carried by member **58** and a reference line **63** carried by the yoke structure. To this structure is attached a pointer **65** which a rocking or rotation of the structure causes to move along a stationary scale **66**. Also affixed to the structure is a graduated balancing arm or bar **67**, along which a slidably mounted weight **68** may be moved in either direction from the central position in which the weight is illustrated.

In preparing the equipment for operation, the sample **50** is so attached to the supporting member **58** that the reference direction of rolling indicated by the dotted arrow **70** of the sheet material from which the sample is stamped is aligned with the 90° point on the named scale. When this member is set, as illustrated, with the 0° point on its scale coinciding with the supporting yoke structure reference mark **63**, the magnetic field will, in passing from one pole piece of the testing apparatus to the other, flow through the sample in a direction which coincides with that in which the sample material was rolled. A shifting of the sample carrying member in the yoke structure then displaces the sample rolling direction from that of the flux flow by an angle represented by θ in Fig. 13, which angle is directly indicated by the carrying-member scale. The intensity of the magnetic field produced by the testing apparatus is sufficiently high to assure that the flux will always flow through the sample in the same direct path, which, in the illustrated equipment, is horizontal.

For a material of the completely non-fibered or randomly oriented grain variety in which the magnetic characteristics are the same in all directions, there will be substantially no torque set up in the circular test specimen by the passage of flux therethrough, regardless of its rotational position in the apparatus, and the pointer **65** will remain in the indicated mid position as long as the balancing weight **68** is at the center of the graduated arm **67**. However, when the sample **50** is of the highly fibered or preferentially oriented grain variety, which possesses preferred directions of easy magnetization, there will be certain rotational ranges where appreciable values of torque are set up by the flow of magnetic flux therethrough.

The apparatus of Fig. 12 measures for as many rotational settings as it is desired to observe this torque in terms of the position along the scale arm **67**, to which the weight **68** must be moved, to bring the pointer **65** to the illustrated central position. Positions of the weight along the arm **67** on one side of its zero or central point thus indicate positive torques, while positions on the opposite side indicate negative torques.

One explanation for the production of torque in the specimen is the action of the forces within the crystals which tend to tie the direction of magnetization with certain of the crystal axes. The torque which results from the action of these forces is the rate of change of magnetic energy with the angle of displacement of these axes from the actual direction of flux flow.

In Fig. 14 there is illustrated the results of tests made upon samples taken from a sheet of 3.25% silicon steel prepared by the strain-annealing process of the present invention. Curve **72** applies to the material after subjection to one of the cold rolling operations, while curve **74** applies to the same material after receiving a subsequent annealing treatment. In both cases the zero point on the horizontal scale of the curves indicates the direction of rolling of the particular material from which the specimen **50** was taken.

It will be noted that before the material is annealed, displacement of the magnetization from the direction of rolling in a given direction causes positive values of torque to be set up in the specimen, the maximum of which in the first half cycle of the curve **72** is indicated by T_{m1}. However, after the material is annealed, comparable displacements between the magnetization and rolling directions set up in the specimen negative values of torque, the maximum of which during the first half cycle of the curve **74** is indicated at T_{m2}. It has been observed that in the case of silicon steel of the character under discussion, such a reversal of torque is an indication of a change in the preponderance of grain orientation from the first type discussed in connection with Figs. 6 to 8, inclusive, to the second type.

Consequently, if for each of a plurality of tests made on the material at different points in the program of treatment the values of the maximum positive torques T_{m1} observed are plotted above a horizontal reference line and those of the observed negative torques T_{m2} are plotted below this line there will result a curve of the general shape depicted in Fig. 8, the significance of which curve has already been discussed.

It will be seen that the described process of the present invention affords means for producing ferro-magnetic material in sheet-like form having characteristics far superior to those attainable through the use of any process previously known. Furthermore, the basic idea of producing highly preferred crystal orientation in magnetic materials to enhance their permeabilities at high flux densities, is capable of broad application and may be extended to materials of the face-centered cubic crystal variety typified by nickel with comparably beneficial results. In such application, however, the different magnetic characteristics of the individual crystals and their different behaviour when subjected to annealing must, of course, be properly taken into account. By the term "steel" as used throughout the specification,

applicants have referred to iron-base mixtures containing carbon in any desired relatively small amount without regard to whether that quantity is or is not sufficient to produce pearlite on slow cooling. The term as here used thus includes materials in which the carbon content may be of the low order of 0.005% or even less.

Since certain changes may be made in carrying out the above process without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is claimed as this invention:

1. In the method of making ferro-magnetic sheet material containing silicon and having improved electrical properties, the steps consisting of casting a steel ingot of the desired silicon content, reducing by hot rolling to a thickness appreciably in excess of the finished size, reducing to the finished size by a plurality of cold rolling operations each of which effects a reduction in thickness of between 30% and 60% and thereby introduces substantial strains into the material that establish a preferred orientation of the material grains in which the crystallographic axes of easiest magnetization of the sheet material grains is aligned in a direction at an angle to the direction of rolling, in annealing after each of said rolling operations prior to the last at a temperature between 800° C. and 1000° C. to relieve these strains and to establish a preferential orientation of the crystallographic axes of the sheet material grains which offer the least resistance to magnetization in a direction substantially coincident to that in which the sheet was rolled, in cooling the sheet after each anneal prior to the next rolling operation, and in a final anneal after the last rolling operation for a period of several hours at a temperature between 1100° C. and 1300° C.

2. In the method of making ferro-magnetic sheet material containing silicon having improved electrical properties, the steps consisting of reducing the material from considerably in excess of its final thickness to its final thickness by a plurality of rolling operations each of which effects a reduction in thickness of between 30% and 60% thereby introducing substantial strains into the material and following each of said rolling operations by a heat treatment, each heat treatment except the last being at a temperature of between 700° C. and 1100° C. which relieves these strains and orients the crystallographic axes of the sheet material grains which offer the least resistance to magnetization in a direction substantially coincident with that in which the sheet was rolled, the final heat treatment being at a temperature of between 1100° C. and 1300° C. for a period of several hours.

3. In a method of making magnetic iron alloy in sheet-like form containing up to 6% silicon and characterized by relatively high permeability and relatively low watt loss, the steps which comprise reducing the sheet to its finished size by a plurality of cold rolling operations each of which effects a reduction in thickness of between 30% and 60% and thereby introduces substantial strains that align the crystallographic axes of the sheet material grains which offer the least resistance to magnetization in a direction at an angle to the direction of rolling, following each of said rolling operations except the last by an anneal at a temperature between 700° C. and 900° C. to relieve the strains and orient the crystallographic axes of the sheet material grains which offer the least resistance to magnetization in a direction substantially coincident with that in which the sheet was rolled, in cooling the material substantially to room temperature after each anneal prior to the next rolling operation, and in following the final rolling operation by an anneal at a temperature between 1100° C. and 1300° C. for several hours.

4. In the method of making ferro-magnetic sheet material containing silicon having improved electrical properties, the steps consisting of reducing to the finished size by a plurality of rolling operations each beginning substantially at room temperature and each of which effects a reduction in thickness of between 30% and 60% thereby to align the crystallographic axes of easiest magnetization of the sheet material grains in a direction displaced by 45° from the direction of rolling, and heat treating after each of said rolling operations except the last at a temperature between 700° C. and 1000° C. to shift the orientation of the grain axes to a desired position which is substantially coincident with that in which the sheet was rolled, in cooling the material substantially to room temperature after each anneal prior to the next rolling operation, and in following the final rolling operation by an anneal at a temperature between 1100° C. and 1300° C. for from 7 to 12 hours.

5. Magnetic material comprising silicon-iron alloy sheet reduced to its final size by a plurality of cold rolling operations, each of which introduces a reduction in thickness of between 30% and 60% and which introduces high intensity strains that align the crystallographic axes of easiest magnetization of the sheet material grains in a direction at an angle to the direction of rolling and subjected to an annealing treatment following each rolling operation at a temperature of the order of 900° C. which relieves the strains and aligns the crystallographic axes of easiest magnetization of the sheet material grains in the direction of sheet rolling and produces a material having relatively low watt losses and high-density permeability characteristics of the high order typified by a permeability of 6400 at a flux density of 16,000 gausses.

6. Magnetic material comprising silicon-iron alloy sheet reduced to its final size by a plurality of cold rolling operations, each of which effects a reduction in thickness of between 30% and 60% and which introduces high intensity strains and so orients the alloy material grains as to align the crystallographic axes of easiest magnetization thereof at an angle of substantially 45° from the direction of rolling and subjected to an annealing treatment following each rolling operation at a temperature of the order of 900° C. which relieves these strains and so orients the alloy material grains as to align the said axes of easiest magnetization in the direction of sheet rolling and produces a material having high density permeability characteristics of the order typified by a permeability of 6400 at a flux density of 16,000 gausses and watt losses of the order typified by 0.33 watt per pound in a sheet 14 mills thick when subjected to a 60 cycle flux of 10,000 gausses maximum density.

7. Magnetic material of silicon iron alloy sheet reduced to its final size by a plurality of rolling operations carried on at substantially room temperature to effect a reduction in thickness of between 30% and 60% and which introduces high intensity strains that align the crystallographic axes of easiest magnetization of the sheet mate-

rial grains in a direction at an angle to the direction of rolling and subjected to an annealing operation following each rolling operation carried on at a temperature of between 800° C. and 1000° C. to relieve the strains and align the crystallographic axes of easiest magnetization of the sheet material grains in the direction of rolling and subjected to a final anneal of from 7 to 12 hours at a temperature of between 1100° C. and 1300° C. producing a material having relatively low watt losses and high density permeability chacteristics of the high order typified by a permeability of 6400 lines at a flux density of 16,000 gausses.

ALBERT A. FREY.
FRANCIS BITTER.