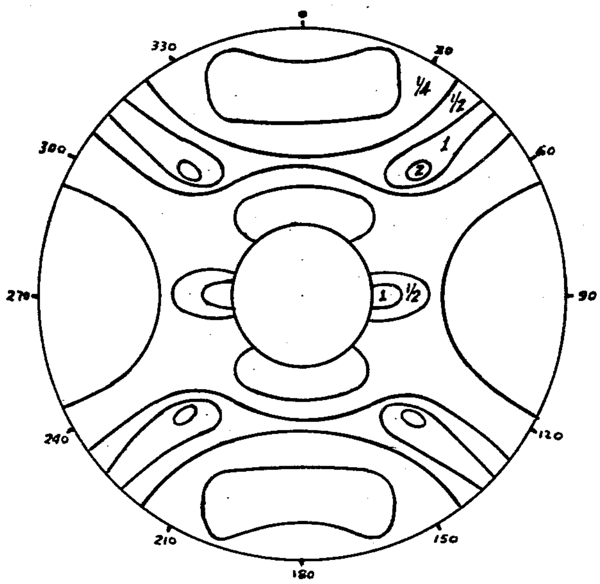

FIG. 1.

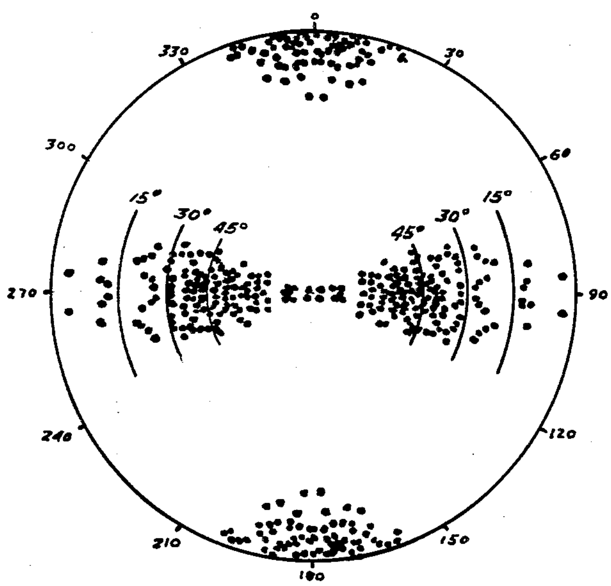

FIG. 2.

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MANUFACTURE OF SILICON-IRON HAVING CUBIC TEXTURE

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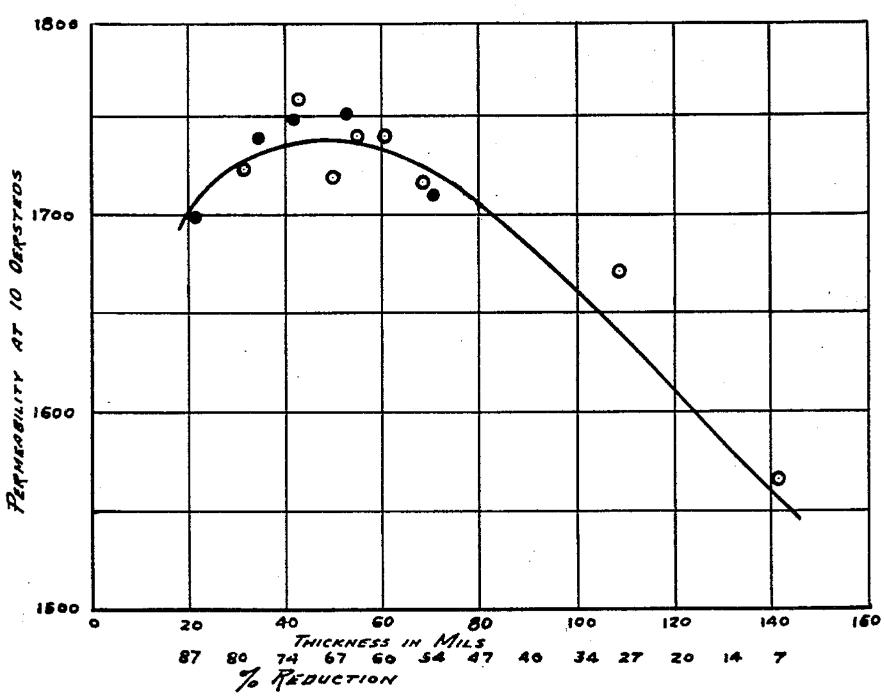

FIG. 3.

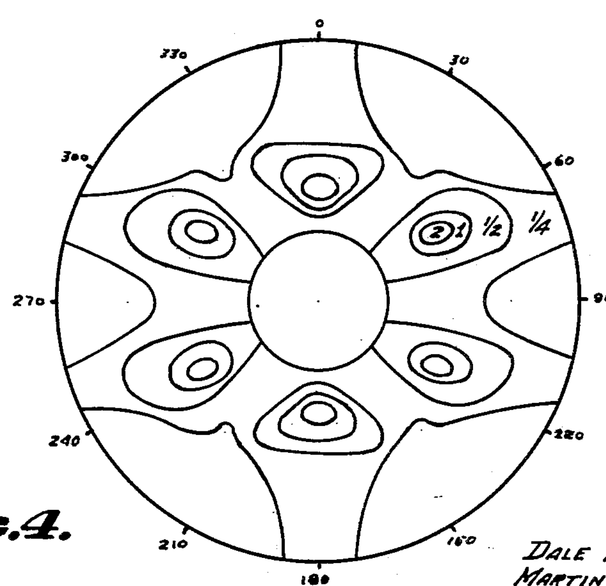

FIG. 4.

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MANUFACTURE OF SILICON-IRON HAVING CUBIC TEXTURE

Filed Oct. 13, 1961 4 Sheets-Sheet 3

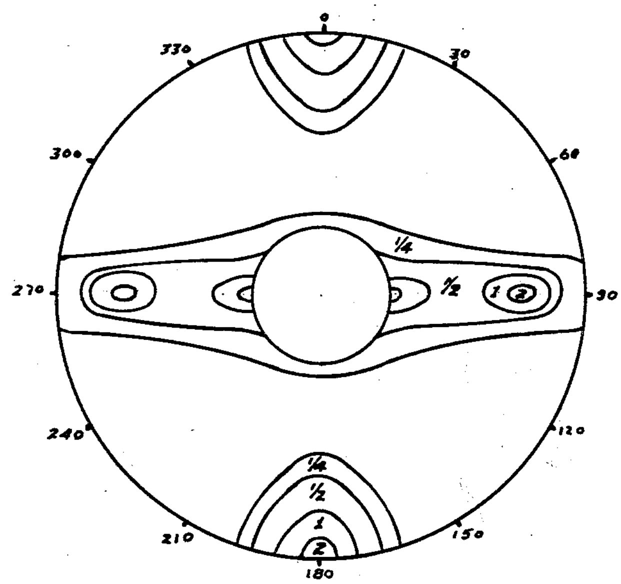

FIG. 5.

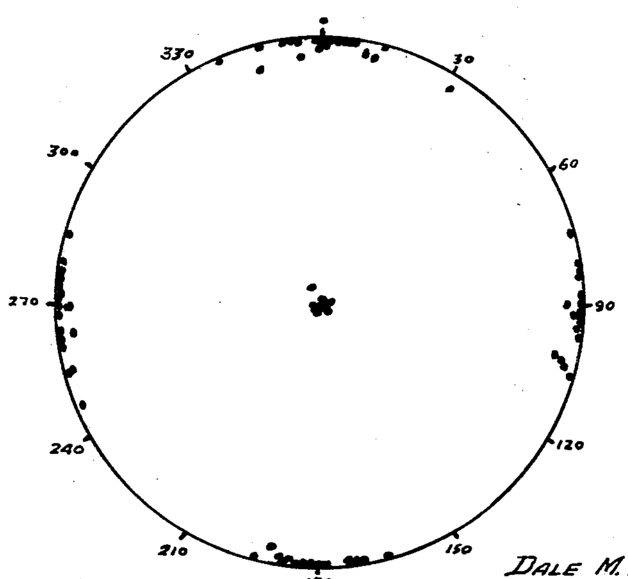

FIG. 6.

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HOT ROLL

HEAT TREAT TO RECRYSTALLIZE- AND OPTIONALLY REMOVE SOME CARBON

COLD ROLL AT LEAST ABOUT 55% TO OBTAIN ORIENTATION TYPIFIED BY FIGURE 1

BOX ANNEAL AT 2000°-2350°F IN HYDROGEN TO REDUCE TOTAL OXIDES TO .005% OR LESS, CARBON TO .010% OR LESS, AND SULFUR TO .005% OR LESS —— ORIENTATION AFTER ANNEAL SHOWN IN FIGURE 2

COLD ROLL AT LEAST ABOUT 55% TO OBTAIN ORIENTATION TYPIFIED BY FIGURE 4

BOX OR OPEN ANNEAL AT 1300°-1700°F. IN NON-OXIDIZING ATMOSPHERE TO CAUSE PRIMARY RECRYSTALLIZATION (FIGURE 5), AND AT 2000°-2300°F. TO OBTAIN SECONDARY RECRYSTALLIZATION AND CUBIC ORIENTATION (FIGURE 6)

Fig. 7

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United States Patent Office 3,130,094
Patented Apr. 21, 1964

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3,130,094
MANUFACTURE OF SILICON-IRON HAVING CUBIC TEXTURE

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Filed Oct. 13, 1961, Ser. No. 145,540
13 Claims. (Cl. 148—111)

This is a continuation-in-part of the copending application of the same inventors, Serial No. 819,589, filed June 11, 1959, now abandoned, and bearing the same title as this application.

It has hitherto been understood that a silicon-iron sheet stock in which the grains have preponderantly an orientation which can be described as a (100)[001] orientation by Miller's indices has a number of useful advantages in the electrical arts. In particular, and depending upon the perfection of the orientation, such a sheet stock may have an unusually high permeability in the straight grain or rolling direction coupled with a high permeability in the cross grain direction, so that its utility is not confined to the manufacture of magnetic core structures in which the primary direction of the magnetic flux must always be parallel to the rolling direction.

It has also been understood that if a sheet of silicon-iron containing a number of grains or crystals having the (100)[001] orientation is, under the proper circumstances, subjected to secondary recrystallization at high temperature, the grains having the designated orientation (frequently referred to as "cubic texture") can be made to grow at the expense of grains in the silicon-iron having other orientations to produce a product in which the greater part of the grains exhibit the cubic texture.

It is necessary to provide a material for secondary recrystallization which will have a reasonable number of grains oriented in the (100)[001] position, or close to it as hereinafter set forth. In the copending application of the inventors here entitled Oriented Silicon-Iron and Process of Making It, Serial No. 816,889, filed May 29, 1959, there is described a method of producing a stock having desirable qualities for the secondary recrystallization treatment in which the starting material is a silicon-iron stock having a high degree of cube-on-edge texture, i.e., a (110)[001] orientation by Miller's indices. In accordance with the teachings of that application, the material is carried through a series of relatively well-defined derivative orientations to produce a material which, after a primary recrystallization, will be in a condition to produce a cubic texture material in a secondary recrystallization treatment.

The procedure of the said application has the advantage of producing a very large number of grains which can grow in the desired cubic texture upon secondary recrystallization, so that an end product may be produced in which the grains are relatively small. But the procedure also has certain disadvantages.

In the first place, the starting material is the equivalent of commercial oriented silicon-iron having the cube-on-edge texture. This means it will already have been subjected to a series of expensive treatments involving cold rollings and intermediate and final anneals. The cost of the steps outlined in the said copending application thus becomes additive to the cost of preparing the starting material.

In the second place, while there is no necessary limitation on the thickness or gauge of the final product, it will be clear that if the steps of the said copending application are applied to a material of commercial gauge and cube-on-edge character, the final product must of necessity have a very light gauge. Cube-on-edge stock may be produced at heavier than ordinary commercial gauges; but procedures for forming the cube-on-edge materials at gauges very substantially heavier than ordinary commercial gauges for oriented silicon-irons present practical difficulties and are likely to be lacking in efficiency.

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It is an object of the present invention to provide a method of making a product suitable for transformation to the cubic texture in which the starting material may be a hot rolled strip or sheet of conventional gauge, and in which no more than a total of two cold rolling treatments is required.

It is an object of the invention to provide in a simpler and cheaper fashion a finished product characterized by cubic texture.

It is an object of the invention to facilitate the manufacture of cubic textured silicon-iron sheet stock of common gauges such, for example, as are used in transformers and electrical machinery, which gauges may be considered as ranging from about 10 to 14 mils or thicker.

It is both an object of the invention to facilitate the manufacture of cubic textured silicon-iron sheet stock having high permeabilities in the rolling direction and at right angles thereto, such stock being especially valuable in the making of transformer cores with stamped, angularly shaped laminations, but also by a modification of procedure to facilitate the manufacture of a stock having a somewhat lesser permeability in the rolling direction but somewhat better permeabilities in intermediate directions, the last mentioned stock having certain advantages for use in rotating electrical machinery.

These and other objects of the invention, which will be set forth hereinafter or will be apparent to one skilled in the art upon reading these specifications, are accomplished by that series of treatments of which a preferred embodiment will now be set forth in detail. Reference is made to the drawings accompanying this specification, wherein:

FIG. 1 is an X-ray pole stereogram of the [200] poles of a silicon-iron stock which has been cold rolled from a thickness of about .150 inch to about .050 inch.

FIG. 2 is an optical pole figure of the orientation produced by annealing the material of FIG. 1 for 100 hours at 2200° F.

FIG. 3 is a chart in which the permeability of material having the general orientation shown in FIG. 2 is plotted against the degree of cold reduction. The data in FIG. 3 were taken from two heats as indicated by the solid circles and the open circles. The reduction scale shows thickness in mils for the material of FIG. 2, there being another horizontal scale marked in percentages of reduction, applying as well to other thicknesses.

FIG. 4 is an X-ray stereogram of the [200] poles of the material of FIG. 2 after it has been cold rolled in a second operation from a thickness of .050 inch to a thickness of .012 inch.

FIG. 5 is an X-ray stereogram of the [200] poles of the material of FIG. 4 after it has been subjected to a primary recrystallization by being box annealed at a temperature of about 1400° F. for about one hour.

FIG. 6 is an optical pole figure of a final orientation produced by subjecting the material of FIG. 5 to secondary recrystallization as hereinafter taught.

FIG. 7 is a flow sheet diagram of the steps of a typical process embodiment of the invention as applied to silicon-iron containing substantially 2.5% to 4% silicon and not more than 0.040% total oxides.

In the X-ray stereograms constituting FIGS. 1, 4 and 5, small numbers indicate the intensity in "times random."

Since in a proper secondary recrystallization treatment grains having the (100)[001] orientation, and orientations approximating it, will grow at the expense of grains having substantially different orientations, the material which is to be subjected to the secondary recrystallization should have certain characteristics both as to orientation and as to chemistry, which will hereinafter be outlined.

In the provision of a material suitable for secondary recrystallization to produce a material having the most perfect cubic orientation, it is necessary that the product contain a substantial number of well dispersed crystal nuclei having a cube face less than 5° and preferably less than 2° of angularity to the surface of the sheet stock and that as to these grains, the orientation of the cube edges should be such that most of the edges are parallel to the rolling direction. It is preferred that at least 75% of the cube edges lie within 20° of the rolling direction. It is not possible to state a limiting proportion of the cubic grains which should lie in the orientation limits just described, since the ultimate cubic texture will be a result of a secondary recrystallization in which grains so oriented grow at the expense of grains having substantially different orientations, and single crystal materials are entirely possible.

The greater the number of grains lying in or substantially in the cubic orientation in the sheet stock at the start of the secondary recrystallization, the smaller will be the grain size of the final product. This is advantageous from the standpoint of core loss. Also the desired cubic texture will be attained somewhat more easily, and in a shorter length of time when the material at the start of the secondary recrystallization contains a large number of properly oriented grains.

As will be evident from the copending application referred to above, a product containing a reasonable number of properly oriented nuclei can be produced from a material having cube-on-edge orientation by a procedure involving tilting the cube faces to varying angles transversely of the direction of rolling to such an extent that there will be a number of the grains having their cube faces tilted into parallelism with the surface planes of the sheet or lying within 5° of that position.

The procedure of the said copending application, in which a silicon-iron having a high degree of the cube-on-edge orientation is used as a starting material, is advantageous in that the cube edges will have been aligned quite perfectly in the rolling direction. This alignment is referred to as "azimuthal orientation." When a material has a high degree of such orientation, the problem becomes one of tilting the cube faces with respect to the planes of the sheet stock surfaces until a reasonable number of the grains attain the cubic orientation.

If a starting material could be obtained which was characterized by perfect cube-on-edge texture, two stages of cold reduction would in theory be necessary, the first yielding a tilting of the cube faces about 22½° from the cube-on-edge position, and the second yielding a number of grains tilted into or close to the cubic texture position. This is essentially what is done in the practice of the copending application referred to above, it being kept in mind that no orientation is ever perfect in a polycrystalline material.

It has been found in the practice of the present invention that it is not necessary to start with a material having a high degree of cube-on-edge orientation to attain a material having an acceptable azimuthal orientation. On the contrary, under carefully controlled conditions, a satisfactorily high degree of azimuthal orientation can be achieved by subjecting a hot rolled material to a single stage of cold rolling and an intermediate anneal, and this azimuthal orientation can be caused to persist through other treatments into the material ready for the final secondary recrystallization. In the practice of this invention there is a second cold rolling stage as will be set forth; but the azimuthal orientation does not appear to be very greatly improved, if improved at all, by the second cold rolling and an ensuing primary recrystallization. Rather one problem in the manufacture of the most highly oriented material is the preservation during the second cold rolling treatment of the relatively high degree of azimuthal orientation which has been produced in the first cold rolling treatment. The second rolling treatment, however, is important in attaining the desired final tilt of the cube faces into parallelism or substantial parallelism with the surfaces of the sheet stock.

It has further been found that when a hot rolled silicon-iron stock is subjected to a cold rolling treatment, and an intermediate primary recrystallization, a material will be obtained having not only an acceptable degree of azimuthal orientation but also having a very substantial number of grains with cube faces tilted to an angle of 32° or less from parallelism with the sheet surfaces. If a number of the grains after the first cold rolling and an intermediate anneal have such a tilt, then a second cold rolling and primary recrystallization can be depended upon to produce an adequate number of grains having the cubic texture within the limits set forth.

The orientation obtained after the first stage and intermediate anneal is not a true cube-on-edge orientation although a large number of the grains will be in that position. It may be characterized as an imperfect cube-on-edge orientation; and the less perfect it is, so long as there is a high degree of azimuthal orientation, the greater will be the number of grains having the cubic texture in the material at the start of the secondary recrystallization. The effect of the second cold rolling treatment and the resulting primary recrystallization will be to produce a substantial number of grains in which the cube faces are either parallel to the sheet surfaces or lie within 5° of such parallelism, and in which the cube edges are substantially aligned in the rolling direction. It will be understood that some deviation or scatter in azimuthal orientation is tolerable, and in one phase of the invention, as hereinafter set forth, some scatter in azimuthal orientation is deliberately permitted.

The silicon-iron to be treated is preferably melted and refined in the open hearth furnace or equivalent treatment apparatus, and it is a commercial advantage of the process that it may be so melted and refined. A vacuum melting technique may, however, be employed if desired.

The composition of silicon-iron should be substantially as follows:

Silicon	2.5% to 4% with 2.90% to 3.30% preferred.
Manganese	.03% to .15%.
Sulfur	.015% to .030%.
Carbon	.015% to .030%.

The balance will be iron with such impurities as are normal in the manufacture of high grade silicon-iron. However, the product, if it contains any aluminum, should not contain aluminum in excess of about .004%. The material should be as clean as possible. A total oxides content over about .040% is undesirable. Materials which form oxides that are not reducible in hydrogen having a −50° dew point at 2200° F. should be at a minimum and preferably below .004%. The analysis given is that for the hot rolled material as distinguished from a ladle analysis. The manganese and sulfur contents are important and care should be taken to maintain them within the ranges set forth whether the material be vacuum melted or air melted.

The silicon-iron material is preferably hot rolled from a high slab or ingot temperature because this improves the quality of the orientation. The precise gauge to which the material is hot rolled may be varied depending on the desired final thickness. As an example, for the production of a final product of 12 to 14 mils in thickness, the silicon-iron may be hot rolled to a thickness of about .150 inch. The hot rolled gauge of the material can be varied in view of the desired final gauge, and the desired percentages of reduction in the two cold rolling stages, as will hereinafter be explained.

The hot rolled material is then given an initial anneal which may be a box anneal at about 1400° F. in air for a total time of 24 hours. An open anneal in air may be substituted for the box anneal; but if this is done a higher temperature should be used, namely a temperature of about 1800° F. with a soaking period of several minutes. A box anneal, if employed, may be carried on with the hot mill scale still on the surfaces of the stock, and it may be followed by a brief open anneal in air to facilitate pickling. The hot rolled material will in any event be pickled to provide a clean surface.

One of the actions which will occur during this initial anneal is a reduction of carbon. The exact amount of carbon so removed is not controlling at this point since additional carbon may be removed at the intermediate anneal hereinafter described.

After a thorough cleaning of its surfaces, the stock is cold rolled in a first stage with a reduction of about 55% to 80%, an optimum reduction being about 67%. In an exemplary procedure, the hot rolled stock having the aforesaid thickness of about .150 inch may be cold rolled to a thickness of about .050 inch, but these values are not limiting. In another exemplary procedure hot rolled material having a thickness of about .110 inch may be cold rolled in this first stage to about .033 inch, which is a reduction of about 70%.

FIG. 1 is illustrative of the orientation of the stock after the first cold rolling. It will be seen that a start has been made toward azimuthal orientation and also that a tilting of the cube faces with respect to the planes of the sheet stock is apparent.

After this reduction the stock is subjected to a high temperature heat treatment or box anneal in hydrogen. By "high temperature" is meant a temperature substantially within the range of 2200° F. to 2350° F. The annealing is practiced for a considerable length of time, i.e., about 30 to about 90 hours at the high temperature. A number of actions occur during this anneal. The carbon, unless previously reduced to a minimal value, will be reduced to 0.010% or less. Oxide inclusions in the metal will also be reduced; and after this heat treatment, the total quantity of such oxides should preferably be less than about .005%. If the oxides in the starting material are substantially lower than about 0.04%, a somewhat lower temperature may be employed for this intermediate anneal, i.e., a temperature down to about 2000° F. The total quantity of oxides in the starting material will be lower if the material has been manufactured by the vacuum melting technique, and products so produced may be used in the process of this invention. However, as indicated above, an advantage of the invention is that it may be applied with entire success to air melted silicon-iron as produced, for example, in the open hearth furnace. The intermediate anneal also has a function in lowering the sulfur content of the silicon-iron. At the end of the intermediate anneal the sulfur content will preferably have been lowered to .005% or less, thereby diminishing the time necessary for accomplishing secondary recrystallization in the final anneal.

From the above, it will be evident that the temperature of the intermediate anneal and the time at temperature, may be varied depending upon the amount of impurities in the stock. If the material is initially low in impurities as the result of melting and refining processes practiced upon it, a box anneal at a temperature of about 2000° F. for a few hours may be sufficient. Normally, however, open hearth stock is relatively high in oxides, and such stock may require a temperature of about 2200° F. or higher for times up to 90 hours to reduce the impurities to the desired level.

A recrystallization of the metal will of course occur in this intermediate anneal. As indicated in FIG. 2, the orientation and texture of the metal following the recrystallization may be regarded as an imperfect form of cube-on-edge texture, in which the cube edges are reasonably aligned in the rolling direction, and in which a considerable number of the grains have their cube faces lying at an angle of 45° to the plane of the surface of the sheet. But it will be seen that there is a very substantial spread in the lateral tilt of the grains and in particular that an appreciable number of the grains have cube faces lying at an angle of less than about 32° to the planes of the surfaces of the sheet. The optical pole figure, forming FIG. 2 of the drawings hereof, shows that some of the grains have even attained a condition of cubic orientation. In this figure certain semi-circular lines, so marked, have been used to indicate positions of tilt of 45°, 30° and 15°.

Reference is now made to FIG. 3 which shows diagrammatically the result of a first cold rolling stage at different percentages of reduction on the permeability of the product after the intermediate anneal. The permeability is important because it indicates among other things the degree of perfection of the azimuthal orientation. While the precise shape of the curve may vary with different formulae and different starting thicknesses, it will be seen that the highest permeabilities in the first stage of cold rolling are attained with cold rolling reductions of about 55–80%. The skilled worker will also recognize that the permeabilities are not as high as those obtainable in finished commercial grade silicon-iron stocks having the most perfect cube-on-edge orientation. Nevertheless, a relatively high degree of straight grained permeabilities is attained at the first stage cold rolling reductions hereinabove taught, and is indicative of a relatively high degree of azimuthal orientation.

In the making of stock having the most nearly perfect cubic orientation, the next step is a second cold rolling in which a reduction of about 75 to 90% is effected. For example, if the thickness of the sheet at the time of the intermediate anneal was .050 inch, it may be reduced in the second cold rolling treatment to a thickness of .012 inch, assuming that this is a desired final gauge. The thickness figures just given are, of course, illustrative but not limiting.

The texture immediately following the second stage cold rolling will approximate that shown in FIG. 4. When a material having such texture is subjected to a primary recrystallization it assumes the texture shown in FIG. 5. As will be seen from the last mentioned X-ray stereogram the material is still characterized by a high degree of azimuthal orientation and a tilting of a substantial number of the grains into the positions responding to the (100)[001] orientation, or to positions close to it.

In order to obtain the X-ray pole stereogram which is FIG. 5 hereof, some of the material after the second stage of cold reduction was subjected to a box anneal at 1400° F. for one hour to effect a primary recrystallization but without producing substantial secondary recrystallization. It does not violate the spirit of this invention to subject the silicon-iron after the second stage cold rolling to a primary recrystallization only, leaving the secondary recrystallization to be carried on at some subsequent time. Thus it would be possible, although somewhat unusual, to effect a primary recrystallization at this stage and then sell the material. The customer could stamp laminations from such a material and carry on the secondary recrystallization thereafter, with the laminations in stacked form and separated by a suitable annealing separator.

A more usual operation will be to carry on the primary and secondary recrystallization as a part of the same heat treatment. The skilled worker in the art will understand that primary recrystallization occurs quite rapidly at a relatively low temperature, say 1300° F. to 1700° F. The secondary recrystallization takes time and occurs at temperatures roughly within 1900° F. to 2300° F. Consequently the primary recrystallization can be considered to occur and will normally be considered to occur while the material is being heated up to the temperature for secondary recrystallization. Thus a box annealing of the material in dry hydrogen (i.e., hydrogen having a —50° dew point at about 2200° F.) is practiced on the silicon-iron stock usually but not necessarily in the form of stacked sheets spaced by a suitable annealing separator.

Instead of hydrogen, inert atmospheres like helium or argon may be employed or vacuum annealing may be practiced. Secondary recrystallization is facilitated by a high degree of purity in the material and by various other factors. One of these is surface smoothness, which may be attained in accordance with the teachings of the copending application of Dale M. Kohler, one of the inventors here, entitled Procedure for Secondary Recrystallization, Serial No. 824,915, filed July 6, 1959, by finishing the material in the second stage of cold rolling with polished rolls.

The final heat treatment is preferably carried on in accordance with the teachings of the copending application of one of the inventors herein, Dale M. Kohler, and John M. Jackson entitled The Production of Oriented Silicon-Iron Sheets by Secondary Recrystallization, Serial No. 813,289, filed May 14, 1959. The last mentioned application teaches in essence the use of an atmosphere, in the final heat treatment, of hydrogen or a non-reactive gas such as argon or helium, which atmosphere contains a very small amount (e.g., about 20 to 250 parts per million of hydrogen sulfide), of a highly polar compound such as hydrogen sulfide, sulphur dioxide, an oxide of carbon, or a mixture of these. The highly polar compound is believed to be absorbed or adsorbed on the crystal planes at the surfaces of the sheet stock so as to satisfy the positive unsatisfied charges there, the result being a shifting of the energies of crystals of differing orientations in such a way that the (100)[001] orientation becomes the lowest energy orientation by a substantial amount, making for a more positive and complete cubic texture orientation in the stock. The tendency toward secondary recrystallization in the cubic texture when the anneal is carried on as just described is so strong that in many instances an open, strand or continuous anneal may be used. The usual practice, however, will be a box anneal with a soaking time of at least several hours at the highest temperature, a temperature of 2000° F. to 2300° F. being preferred. Excellent results are attained at a temperature of about 2200° F.

Example

A silicon-iron material containing 3.21% silicon was hot rolled to a thickness of .150 inch and box annealed at 1400° F. in air for a total time of 24 hours. It was then cold reduced to .050 inch and given an intermediate box anneal in hydrogen at a temperature of about 2200° F. for a time of about 80 hours in an atmosphere of dry hydrogen. Thereafter it was cold reduced to a thickness of about .010 inch and then subjected to a box anneal in dry hydrogen (−50° dew point at 2200° F.). An annealing separator was used consisting essentially of magnesia in a thoroughly dehydrated state and containing a minute trace of sulphur. The anneal was at a temperature of about 2200° F. with a soaking period of 24 hours.

An analysis of the grain directions made by the optical method on 25 randomly selected grains showed a good azimuthal orientation and a tilting of the cube faces toward parallelism with the sheet surfaces as follows:

46%	within 5°
75%	within 10°
92%	within 15°
92%	within 20°
96%	within 25°
100%	within 30°

The permeabilities noted were as follows:

Permeability at H=10 oersteds (straight grain), 1882

Permeability at H=10 oersteds (cross grain), 1880 (estimated to be).

A pole figure obtained by the optical method on this material forms FIG. 6 of the drawings hereof.

As has been indicated the procedure outlined above results in a very high degree of cubic orientation in the final product. The procedure can be depended upon to give straight grain and cross grain permeabilities above 1800. It is possible, however, to follow a procedure as hereinafter outlined, and obtain a less perfect degree of cubic orientation by a similar series of crystal changes. In this modified procedure the initial steps including the preparation of the hot rolled stock, the first cold rolling stage, and the intermediate anneal will be the same as those set forth above. Since the percentage of reduction in the second cold rolling stage will be somewhat less, it becomes possible to start with a lighter hot rolled gauge and arrive at the same final gauge or gauges. By way of example, if the silicon-iron is hot rolled to about .110 inch, and then is given a 70% reduction in the first cold rolling stage and is carried down to about .033 inch, a second cold rolling with a 73% reduction will carry the material down to .009 inch. If the final gauge is to be .014 inch, only a 58% reduction would be required. The modified procedure now being described is not limited to the use of a hot rolled material of any particular thickness; but it will comprise a first cold rolling treatment with a reduction of at least about 55%, and a second cold rolling reduction of at least about 55%. The modified procedure can be depended upon to give permeabilities at least as high as about 1700 in the straight grain and cross grain directions. This permeability makes the stock less desirable for punched laminations in transformer cores; but the stock is less perfectly directional in the plane of the sheet, and therefore has a certain advantage in rotating electrical equipment. This is not to say tha the stock is non-directional. On the contrary, it approximates the character of a true cubic stock, but is characterized by a greater spread in the azimuthal orientation.

In its production, the final primary recrystallization and the ensuing secondary recrystallization will be those described above.

Without wishing to be bound by theory, it is believed that whereas a heavier cold rolling reduction in the second cold rolling treatment is necessary for the preservation of the relatively high degree of azimuthal orientation attained in the first cold rolling treatment, a second cold rolling treatment at a somewhat lower reduction causes the tilting of the grains or crystals so that their faces come more nearly into parallelism with the surfaces of the sheet stock, but at the same time is less effective in preserving the azimuthal orientation. It will be observed that the secondary recrystallization is a surface energy recrystallization. It is believed that under this condition, the grains having their cube faces oriented to parallelism with the stock surfaces (or to within less than 5° of such parallelism) tend to grow in the secondary recrystallization at the expense of grains not so oriented. Thus, the result is a product having a high degree of face-orientation but a somewhat greater spread of azimuthal orientation.

Modifications may be made in the invention without departing from the spirit of it. The invention having been described in certain exemplary embodiments, what is claimed as new and desired to be secured by Letters Patent is:

1. A process for the manufacture of silicon-iron sheet stock having cubic texture, which comprises hot rolling silicon-iron containing substantially 2.5% to 4% silicon and a total oxide content of not more than 0.040% to an intermediate gauge, heat treating the hot rolled stock at a temperature of at least about 1400° F. but not substantially exceeding 1800° F., cold rolling said stock with a reduction of at least about 55%, annealing the said stock at a temperature of about 2200° to 2350° F. in an atmosphere of hydrogen and for a sufficient length of time to recrystallize a substantial number of the grains of said

stock with their cube faces tilted at an angle of less than about 32° from parallelism with the stock surfaces, the stock after the said anneal having oxide inclusions less than about 0.005%, a carbon content not substantially greater than 0.010%, and a sulfur content not substantially greater than 0.005%, again cold rolling said stock with a reduction of at least about 75%, to reduce said stock to final gauge and further to orient the grains therein, and subjecting the cold rolled sheet stock first to a primary recrystallization anneal at a temperature of about 1300° to about 1700° F. in a non-oxidizing atmosphere to produce cubic nuclei having their cube faces tilted at less than 5° to the surface of the sheet stock, said nuclei having also at least about 75% of their cube edges aligned within 20° of the rolling direction, and second to an anneal at a temperature of about 2000° to about 2300° F. in a non-oxidizing atmosphere under conditions to produce secondary recrystallization by surface energy whereby to cause said nuclei to grow by said secondary recrystallization at the expense of grains having substantially different orientations in said sheet stock.

2. The process claimed in claim 1 wherein the heat treatment following said hot rolling is a box anneal at a temperature of about 1400° F. in air.

3. The process claimed in claim 1 wherein the heat treatment following said hot rolling is a continuous anneal at a temperature of about 1800° F.

4. The process claimed in claim 1 wherein the stock is subjected in said first mentioned cold rolling operation to a reduction of substantially 55% to 80%.

5. The process claimed in claim 1 wherein the stock is subjected in said first mentioned cold rolling treatment to a reduction of about 67%.

6. The process claimed in claim 4 wherein the stock is subjected in said second mentioned cold rolling treatment to a reduction of about 75% to 90%.

7. The process claiced in claim 1 wherein said secondary recrystallization treatment is a heat treatment in a non-oxidizing atmosphere containing from 20 to 250 parts per million of a highly polar compound chosen from a class consisting of oxide of carbon, oxide of sulfur and hydrogen sulfide.

8. The process claimed in claim 7 wherein the treatment in said second mentioned cold rolling is finished by rolling the stock between polished rolls with sufficient reduction to produce a smooth polished surface on said stock.

9. The process claimed in claim 6 wherein said stock is hot rolled to a thickness of the order of .150 inch, is cold rolled in the first mentioned cold rolling treatment to a thickness of the order of .050 inch, and is cold rolled in the second mentioned cold rolling treatment to a thickness of the order of .012 inch.

10. A process for the manufacture of silicon-iron sheet stock characterized by cubic texture, which process comprises hot rolling a silicon-iron containing substantially 2.5% to 4% silicon, .03% to .15% manganese, .015% to .030% sulfur and .015% to .030% carbon, and containing no more than about 0.040% total oxide, the balance being iron with such impurities as are normal in the manufacture of high grade silicon-iron, heat treating the hot rolled silicon-iron at a temperature of substantially 1400° to 1800° F. and pickling it, cold rolling the silicon-iron with a reduction of substantially 55% to 80%, heat treating the silicon-iron at a temperature of substantially 2200° to 2350° F. in hydrogen having a dew point of around −50° F. at the lower of said temperatures, for a period of substantially 30 to 90 hours, thereafter again cold rolling the said silicon-iron with a reduction of substantially 75% to 90%, whereby to produce a silicon-iron sheet stock which upon primary recrystallization will contain cubic nuclei having their cube faces tilted at less than 5° to the surface of the sheet stock, said nuclei having also at least about 75% of their cube edges aligned within 20° of the rolling direction, and subjecting said silicon-iron to a heat treatment in which its temperature is raised to substantially 2000° to 2300° F. under conditions to produce a secondary recrystallization of said silicon-iron at the last mentioned temperatures.

11. The process claimed in claim 10 in which the last mentioned heat treatment is carried on in a dry non-oxidizing gas containing about 20 to about 250 parts per million of a highly polar compound chosen from a class consisting of oxides of carbon and surfur and hydrogen sulfide.

12. A process for the manufacture of silicon-iron sheet stock having cube-on-face texture, which comprises hot rolling silicon-iron containing substantially 2.5% to 4% silicon and containing no more than about 0.040% total oxide to an intermediate gauge, subjecting the hot rolled stock to a recrystallization temperature and thereafter to a cleaning, cold rolling said stock with a reduction of at least about 55%, annealing the said stock at a temperature of about 2000° to 2350° F. in a non-oxidizing atmosphere to recrystallize a substantial number of the grains of said stock with their cube faces tilted at an angle of less than about 32° from parallelism with the stock surfaces, said grains having a substantial degree of azimuthal orientation, the said stock after said annealing having oxide inclusions of no greater than 0.005%, a carbon content not greater than about 0.010% and a sulfur content not greater than about 0.005%, again cold rolling said stock with a reduction of at least about 55% to reduce said stock to final gauge and further to tilt the grains therein, and subjecting the cold rolled sheet stock first to a primary recrystallization anneal at a temperature of about 1300° to about 1700° F. in a non-oxidizing atmosphere to produce cubic nuclei having their cube faces tilted at less than 5° to the surface of the sheet stock, said nuclei having also about 75% of their cube edges aligned within 20° of the rolling direction, and second to an anneal at a temperature of about 2000° to about 2300° F. in a non-oxidizing atmosphere under conditions to cause said nuclei to grow by surface energy secondary recrystallization at the expense of grains having substantially different orientations in said sheet stock.

13. The process claimed in claim 12 wherein the second cold rolling effects a reduction of substantially 55 to 75%.

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