

June 14, 1949.

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2,473,156

PROCESS FOR DEVELOPING HIGH MAGNETIC PERMEABILITY

AND LOW CORE LOSS IN VERY THIN SILICON STEEL

Filed Nov. 16, 1944

2 Sheets-Sheet 1

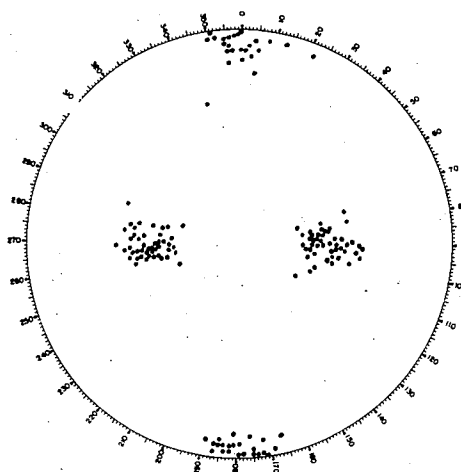


FIG 1

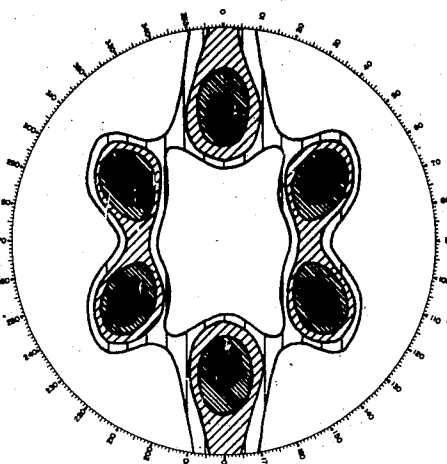


FIG 4

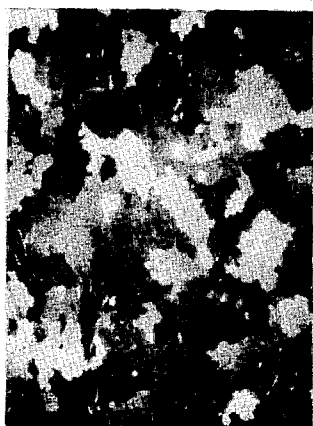


FIG 3



FIG 6

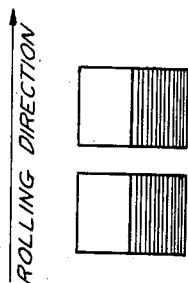


FIG 2

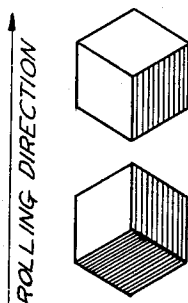


FIG 5

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2 Sheets-Sheet 2

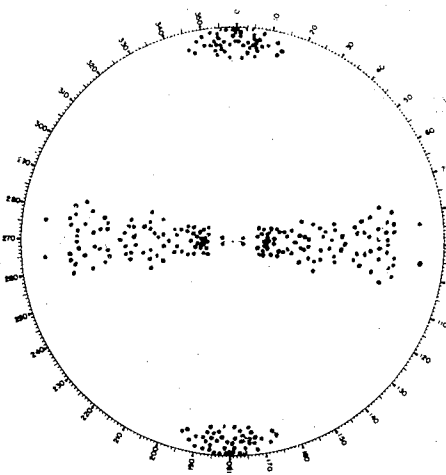


FIG. 7

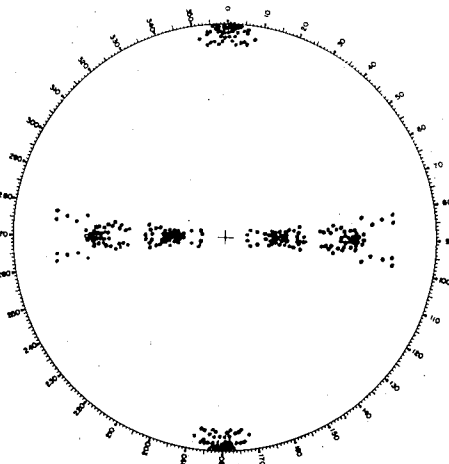


FIG. 10



FIG. 9

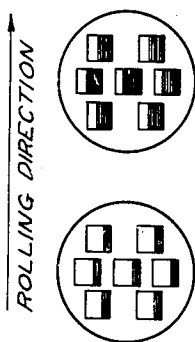


FIG. 8

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2,473,156

PROCESS FOR DEVELOPING HIGH MAGNETIC PERMEABILITY AND LOW CORE LOSS IN VERY THIN SILICON STEEL

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6 Claims. (Cl. 148—12.5)

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Notable progress over the past years has been made in the technology and production of iron-silicon alloys (5 to 5% silicon) toward attaining high permeability and low hysteresis loss at high flux densities. The product has been one characterized by a highly directional permeability, i. e. very high permeability in the rolling direction; and the availability of such a product has brought about radical changes in the design of transformers and other electrical apparatus.

The great majority of electromagnetic applications for such a product (for example, power transformers and the like) has required silicon steel sheets of a thickness lying between 10 and 14 mils. Newer developments, to which great impetus has been given by war needs, especially in electronic, high frequency and other specialized applications, have required the production of favorably oriented silicon steel in thicknesses as low as 1 mil. But efforts to obtain comparable permeabilities and core losses in such thin materials have hitherto been unsuccessful.

The principal object of this invention is the provision of a commercial process for the production of exceedingly thin silicon steel of the order of .5 to 7 mils in thickness, which possesses very low core loss and very high permeability in the straight grain direction, i. e. with the magnetic flux parallel to the direction of rolling.

The ancillary objects of this invention, which will be set forth hereinafter, or will be apparent to one skilled in the art upon reading these specifications, I accomplish in that procedure of which I shall describe certain exemplary embodiments. Reference will hereinafter be made to the drawings wherein:

Figure 1 is a pole figure determined by optical methods for a highly oriented silicon steel of intermediate gauge as respects my product.

Figure 2 is a diagrammatic representation of the crystal orientation in such a steel.

Figure 3 shows the grain condition in a steel suitable for the purpose of my invention, being a photograph at a magnification of approximately two diameters.

Figure 4 is a pole figure determined by an X-ray diffraction pattern of the same steel after being cold rolled with a reduction of 83%.

Figure 5 is a diagrammatic representation of the crystal orientation in such a steel.

Figure 6 shows the elongation of the grains therein prior to recrystallization, being a photograph at a magnification of approximately two diameters.

Figure 7 is a pole figure characteristic of the

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last mentioned steel after recrystallization in an open anneal.

Figure 8 is a diagrammatic representation of the orientation of the crystals therein, being a photograph at a magnification of approximately two diameters.

Figure 9 shows the grain structure of the steel.

Figure 10 is a pole figure characteristic of the steel of Figures 4, 5 and 6 after it has been recrystallized in a high temperature box anneal.

The procedures of any of the following patents: 2,158,065, dated May 16, 1939, in the names of Guernsey H. Cole et al.; 2,287,466, dated June 23, 1942, in the name of Victor W. Carpenter; and 2,307,391, dated January 5, 1943, in the names of Guernsey H. Cole et al., result in the production of silicon steel sheets of transformer gauge in which the grains or crystals have an orientation of the [100] (110) type in the standard notation by Miller's Indices. This notation indicates that the [100] direction of the crystals is parallel to the rolling direction and a (110) plane is parallel to the rolling plane. Considering the crystals as cubes, the notation indicates a cube-on-edge position of the crystals in the plane of the sheet, with parallel edges extending in the rolling direction as indicated in Figure 2 where the rolling direction is shown by an arrow and the rolling plane is the plane of the drawing.

This orientation and the enhanced magnetic properties resulting from it have hitherto been attained in materials of the order of 7 to 25 mils in thickness by the use of cold rolling and annealing treatments in one, two or three stages coordinated with each other in accordance with the teachings of the said patents. The number of parts or stages of cold rolling employed was determined in large measure by the desired final thickness as compared with the available hot rolled starting material.

When very much thinner silicon steel was desired, it was evident that additional cold rolling treatments would be necessary, the gauge range of available hot rolled materials being fixed. But when such additional stages of cold rolling and intervening annealing were introduced into former practices in accordance with the understanding in the art, the expected magnetic quality was not attained. In the production of the thinner silicon steel the crystalline behavior was found to be different from the behavior in the thicker material; and the steel was not responsive in the same way to the same manipulations. In other words, the application of additional rolling and annealing cycles of the prior art type (so

that the thickness of the silicon steel could be carried down to a range between 1 and 5 mils by a total of two, three or more parts or stages of cold rolling), and the application of the known techniques to such a material, did not result in the expected high permeabilities or in the proper crystal orientation.

For example, although a permeability at 10 oersteds of as high as 1500 was sporadically obtained for a material 3 mils in thickness (tested parallel to the rolling direction), no permeability higher than 1400 could be obtained in materials 2 mils thick and thinner. In general, the permeabilities obtainable were of the order of 1450 at 3 mils, 1380 at 2 mils, and 1325 at 1 mil, even under the most favorable annealing conditions. This should be compared with permeabilities of 1650 to 1750 and higher, which are common with oriented materials of 13 mils in thickness made in accordance with the patents listed above. When it is recalled that permeabilities of 1400 for non-oriented alloys of transformer gauge and of similar silicon content is usual, it will be seen that these efforts produced very little orientation of the type giving high permeability in the rolling direction.

If the steel were carried down to very thin gauges in a series of cold rolling stages of the prior art type, the intervening open anneals, the desired type of orientation was not obtained or was obtained only very imperfectly. On the other hand, attempts merely to take the known highly oriented materials and reduce them further by adding further cold rolling and annealing treatments of the same type resulted in a substantial impairment of the desired type of preferred orientation even if obtained at some intermediate point.

Moreover, the core loss values were found to be excessively high for all materials which resulted from these efforts. In attempts to reduce core loss in the thin material, resort was had to open annealing at very high temperatures of from 2000 to 2200° F. or to a box anneal of the thin material at 2200° F. in dry hydrogen. These were difficult procedures; but in spite of them the core loss remained high because of the unfavorable crystal orientation.

By the procedures hereinafter set forth I am able to produce extremely thin silicon steel materials which nevertheless possess high permeabilities and low core losses comparable to those hitherto attained in the heavier gauge electrical sheets. I have found that while a [100] (110) type of crystal orientation attained in a material substantially between 7 and 25 mils in thickness will be modified and changed by subsequent cold rolling practiced on the sheets, the attainment of certain initial qualities in the steel as hereinafter set forth plus the carrying on of subsequent steps under the conditions hereinafter outlined, will produce in the material a modified orientation which still is excellent as respects high and highly directional permeability and low core loss.

While the hitherto known best orientation of the crystals for high permeability is of the [100] (110) type, I have found that there is an equally good modified orientation in which the crystals tend to assume two new positions, each involving a slight rotation about the rolling axis of about 20° in either direction. In this new orientation a [100] direction of each crystal remains parallel to the rolling direction; but the hitherto parallel (110) planes tilt with respect to the rolling plane, and may tilt about the rolling axis in either di-

rection, the angles of tilt of different crystals tending in either direction toward 20° as a mean position.

Thus I have found it possible to take a material having a thickness of 7 to 25 mils or thicker which is already characterized by a high degree of [100] (110) orientation and reduce such a material by cold rolling to exceedingly thin gauges (under the circumstances hereinafter set forth) and achieve high permeability in the rolling direction, although the nature of the orientation will be modified.

In my process, when a material initially oriented in the [100] (110) fashion, having a satisfactorily large grain size and a very high purity, as will be explained, is subsequently cold rolled and recrystallized under the conditions to be set forth, a plurality of small crystals form within the boundaries of the original crystals and assume an orientation which is not the same as the original orientation but is governed thereby to the extent that the derivative crystals assume the orientation last described.

The starting materials to which my invention applies are silicon steels preferably containing from 2.90 to 3.30% silicon, .007% or less of carbon, and .06% to .12% manganese, the remainder being iron, with a total oxide content of .015% or less.

The silicon range is not limiting but may be carried above or below the preferred values without departing from the invention. The preferred range is a conventional working range which, in practice and for high frequency electronic pulse transformers, is limited on the low side by the poorer core loss resulting from low silicon (low resistivity), and on the high side at about 4.5% by the mechanical difficulties encountered in cold working high silicon steel at the present stage of the rolling art. Again, if the silicon content is below 2%, an anneal at a temperature sufficiently high to purify the material properly may cause an allotropic transformation destructive of the initial orientation essential to the process.

The carbon content is important because of considerations of core loss and ductility and for other reasons hereinafter set forth. Increasing carbon contents in the final material are progressively inimical to desirably low core loss values, while at the same time carbon contents above .007% decrease ductility in my starting material when box annealed in a reducing atmosphere at high temperatures. In rolling the starting material to exceedingly thin gauges, brittleness caused by carbon may produce excessive breakage.

The manganese content in my preferred formula has been made low so as to keep magnetically inert materials at a minimum in the thin product. Otherwise it is not limiting on this invention, but is to be considered as to the processing of the silicon steel to produce the [100] (110) orientation at power transformer gauges, as will be understood from a consideration of Patent 2,307,391 referred to above. Under suitable conditions the manganese may be from 0.004 to 0.20%.

The total oxide content is again not limiting, but has been made low in the preferred formula to insure good core loss, and avoid brittleness. Fair success may be had with oxide contents as high as .025%.

In the practice of my invention, I take silicon steel (usually hot rolled) and produce from it a sheet steel around 7 to 25 mils in thickness and

characterized by a high degree of the said preferred orientation, by any of the procedures of the patents referred to above. This forms the starting material for the steps hereinafter taught.

There are important characteristics for this oriented starting material, other than the chemical composition which has already been discussed.

Its actual thickness will depend upon the final gauge and number of parts or stages of cold rolling reduction necessary to attain that final gauge. By the techniques hitherto known, it was difficult to produce a fully oriented material below about 7 mils in thickness. When the thickness of the oriented material exceeds 25 mils, it becomes more difficult to secure the desired perfection of orientation, and the chemical purification (especially carbon elimination) is not so efficient. Within these limits, however, an oriented starting material is produced or chosen in the light of the manipulations required to produce the desired finished gauge.

Thus, the oriented starting material should be 2.5 to 7 times the thickness of the desired end product if one stage of subsequent cold rolling is to be employed. My preferred range is from 3 to 6 times the thickness of the end product. Where the reduction is to be accomplished in multiple stages or parts, the thickness of the oriented starting material will be increased with respect to the final gauge. For example, a two-stage process may be practiced upon a material from 6 to 20 times the thickness of the desired end product. By a stage or part, I mean a cold rolling treatment consisting of any number of cold rolling passes practiced on the material without intervening annealing or recrystallization.

I have already indicated that the orientation of the starting material should be of the [100] (110) type and the perfection of the orientation should be as great as possible. An excellent measure of the degree of perfection of the orientation is the magnetic permeability of the oriented starting material measured parallel to the rolling direction at a magnetizing force of 10 oersteds. A permeability above 1700 corresponds to a high degree of preferred orientation, and I endeavor to obtain such a permeability in my starting pieces, although good end products can be produced from starting pieces having permeabilities as low as 1650 in the rolling direction.

Figure 1 is a typical stereographic pole figure for a satisfactory starting material, the figure being produced from examination of the etched material by reflected light in accordance with known crystallographic techniques, and the plotting of projections of the crystal cube faces. The clumping of points at the 0° and 180° marks indicates the coincidence of a [100] direction with the rolling direction; while the position of the intermediate localized groups along the transverse axis of the figure indicates the coincidence of a (110) plane with the rolling plane. The crystal position is shown diagrammatically in Figure 2.

The oriented starting material should have a large grain size; and while this term is relative, I have found that a grain diameter of from .015 to 15 mm. is a workable range, preferring however, an average grain size of from 1 to 10 mm.

It is essential that the starting material not only possess the desired orientation, but be the product of a high temperature box anneal at

above 2000° F. in a dry hydrogen atmosphere, since high purity and favorably large grain size are developed thereby. It may be stated that the practice of such an anneal at the gauge of the starting material makes it possible to avoid a similar anneal later on in the process where the tight gauge renders such an anneal very much more difficult.

Otherwise, something of a compromise may be effected in grain size. In the confines of each grain of the starting material, the new grains in the ultimate thin material tend to have a size and orientation common to the group, as explained. "Wild" grains tend to form primarily in areas of previous grain boundaries. A high degree of orientation coupled with the largest grain size is thus seen to be the condition most favorable to high permeability in the end product. On the other hand, however, excessively large grain size may, in some rolling procedures, make it difficult to attain flatness when the material is cold rolled to exceedingly thin gauges in one stage.

The processing of the oriented starting material is both simple and flexible. The nature of the rolling instrumentalities employed is not a limitation upon my invention. Under certain circumstances, all of the cold rolling may be performed on the same mill or mills. However, it will be understood that since a product of around 7 to 25 mils in thickness is to be rolled further, the use of a very rigid mill and reduction under tension is indicated, especially where, as is preferred, the material is carried down to the thin gauges in a single thickness. In my commercial practice, I employ a mill of the type set forth in Patent No. 2,170,732, issued August 22, 1939, in the name of Tadeusz Sendzimir. This is a mill with tiny working rolls ultimately supported by means of casters against rigid beams extending across between the mill end frames. But any suitable rolling instrumentality may be employed which is capable of performing the required reductions in silicon steel having widths chosen for commercial requirements.

The amount, per part or stage, of cold rolling reduction practiced upon the oriented starting material is critical in nature. It should be substantially between 60 and 90% and preferably between 67 and 83%, for each part or stage, the higher values being most appropriate for a single part procedure.

Where the cold rolling reduction is to be performed in two stages, I prefer to reduce the piece from 60 to 75% in each part or stage.

The temperature, number of passes, and whether the material is rolled continuously in one direction or back and forth in separate passes, do not appear to influence the results obtained. Excellent results are obtained with mill operating temperatures of the order of 70 to 180° F.

The result of cold rolling the oriented starting material is shown in Figures 4, 5 and 6. Figure 4 is a pole figure constructed from X-ray diffraction patterns, because the cold worked condition of the grains makes it impracticable to apply the reflection technique and plot a pole figure thereby. But the X-ray pattern shows, in the same manner, the orientation of the crystals, which now is that illustrated diagrammatically in Figure 5. The moving toward each other of the dark areas near the 0° and 180° positions shows a tilting of a (110) plane about an axis transverse the rolling axis while

the number and placement of the remaining dark areas show that the tilting has occurred in both senses. The appearance of the grains is shown in Figure 6.

After the cold rolling part or stage, the material is given a heat treatment to effect recrystallization. Either an open anneal or a box anneal may be used with temperatures ranging from about 1200° to about 2200° F. For economic reasons, an open or strip anneal is preferred, and I have found that an anneal of 20 seconds at 1800° F. is satisfactory. By an open or strip anneal, I mean the continuous passage of the strip material through an elongated furnace where the surfaces of the strip are open to the atmosphere of the furnace. The furnace atmosphere itself, however, is controlled; and best magnetic results are obtained in my process by using an atmosphere containing hydrogen.

Where the cold rolling is done in two or more stages, a heat treatment such as has been described should follow each part or stage.

The result of the recrystallization of a material treated as I have described, and having the characteristics shown in Figures 4, 5 and 6, has, I have found, two aspects. First, each original, elongated grain recrystallizes into a large number of smaller crystals forming a group, and behaving similar to each other as though controlled by the original crystal from which they formed. Second, the orientation of the new, smaller crystals has altered again. The pole figure (Figure 7) indicates, by the plotted points near the 0° and 180° marks, that a (110) plane is no longer tilted about an axis transverse the rolling axis, but has righted itself, as it were, in this direction. The other plotted points, substantially along the horizontal axis of the figure indicate a new tilting of a (110) plane about the rolling axis. The clumping of these points indicates that this tilting has occurred in both senses, and measurement shows that the tilting tends toward a mean of 20°. The [100] directions now again coincide with the rolling direction.

I have found that this orientation is one making for a high permeability in the straight grain direction. It is illustrated diagrammatically in Figure 8, while the appearance of the crystals in the steel, after etching, is shown in Figure 9. The perfection of the preferred orientation is very high.

In exemplary practices in accordance with my invention, I first produce, in any of the known ways, a starting material of about 7 to 25 mils in thickness characterized by low carbon, a relatively large grain size, and a high degree of preferred orientation of the [100] (110) type, the characteristics of which have been set forth at length above. As has already been indicated, a final box anneal above 2000° F. in dry hydrogen is of great importance, as improving the orientation and permeability, purifying the metal so as to lower core loss, and producing the desired grain size.

Using the strip steel so produced as a starting material, I now treat it as indicated in the following examples:

EXAMPLE A

A single-stage treatment for silicon steel 5 mils thick

An oriented 3% silicon steel 13 mils thick, as a starting material, having a permeability, measured parallel to the rolling direction, of 1740 at a

magnetizing force (H) of 10 oersteds, and with grains 1 mm. in average diameter, was cold rolled in one or more passes to a thickness of 4.0 to 5.0 mils. It was open annealed for 20 seconds at a temperature of 1850° F. The properties of this material, measured parallel to the rolling direction and obtained after a strain anneal at 1450° F. and measured in accordance with Am. Soc. for Testing Materials test procedure A-34 were:

Permeability for H=10----- 1690
Core loss at 10 kilogausses and 60 cycles
per second -----watts/lb-- .26

EXAMPLE B

A single-stage process for silicon steel 2 mils thick

An oriented 3% silicon steel 12 mils thick produced as outlined above, having a permeability at H=10 of 1740 and with grains 1-4 mm. in average diameter was cold rolled in several passes to a thickness of 2.0 mils and was then open annealed for 20 seconds at 1800° F. in a dry hydrogen atmosphere. The properties of this material, tested parallel to the rolling direction after strain annealing at 1450° F. were:

Perm. H=10 ----- 1710
P 10/60 -----watts/lb-- .36

EXAMPLE C

A two-stage process for making silicon steel 1 mil thick

The same starting material as in Example A was cold rolled in one or more passes to a thickness of 3.5 mils and was then open annealed for 20 seconds at 1950° F. in a dry hydrogen atmosphere. The material so produced possessed a relatively high degree of orientation and a moderately large grain size. It exhibited a straight grain permeability at H=10 of 1660; and the grain diameter was approximately .06 mm.

It was then cold rolled in one or more passes to a thickness of 1.0 mil and was then open annealed for 20 seconds at 1800° F. It exhibited properties parallel to the rolling direction, obtained after a strain anneal at 1450° F., as follows:

Perm. H=10 ----- 1600
P 10/60 -----watts/lb-- .53

EXAMPLE D

A single-stage treatment for exceptionally high quality silicon steel 1.7 mils thick

An oriented 3% silicon steel 7.5 mils thick, produced as outlined above, having a permeability at H=10 of 1770 and with grains 5-10 mm. in average diameter was cold rolled in several passes to a thickness of 1.7 mils and was then box annealed for 30 hours at 2000° F. in a dry hydrogen atmosphere. The properties of this material, tested parallel to the rolling direction were:

Perm. H=10 ----- 1815
P 10/60 -----watts/lb-- .28

Figure 10 is a pole figure for the material of Example D, and it will be noted that the plotted points on the horizontal axis have become more perfectly grouped at positions indicating a tilting of a (110) plane of about 20° in both directions or senses transverse to the rolling direction.

In these examples the straight grain properties were measured after a strain anneal because a strain anneal is incident to the making of suitable finished transformer cores, so that the per-

meability after such a treatment is the important permeability.

These examples are not limiting, but are generally characteristic of my commercial practice. Variations in the procedure may be made as will be understood.

The principal importance of this invention lies in the fact that it has enabled me to produce high permeability, highly directional silicon steel of exceedingly thin gauge, whereas this has not been possible in the past.

One of the great advantages of my method lies in the simplicity and ease of control of the processing following the production of the oriented starting material. The highly oriented, large grain, low impurity material at around 7 to 25 mils in thickness can readily be tested for its suitability for rolling to light gauges. Within reasonable limits, the magnetic quality of the final product at from .5 to 5 mils in thickness is predetermined by the properties of the oriented starting material. Processing variables leading to the formation of the oriented starting material do not control the nature of the subsequent processing steps, and affect the nature of the final thin gauge product only insofar as they influence the readily evaluated properties of the starting material.

It should be noted that very large cold rolling reductions are practiced upon the oriented starting material without harming the magnetic properties of the final product. For example, properly oriented silicon steel of 13 mils thickness may be directly rolled to a product 2 mils thick without intermediate annealing, and this is a reduction of 85%. Yet, the permeability of the final product will be high if the starting material had the proper characteristics. This is contrary to past experience, wherein lower percentages of reduction were employed on relatively unoriented materials and wherein high permeabilities were not obtainable at all in thin materials, but wherein the obtainable permeabilities were found to become less as the percentage reduction per part or stage was increased.

It may also be noted that the large grained oriented material which I produce as a starting material is more easily reduced by cold rolling than are conventional, fine grained materials not so oriented.

That excellent magnetic characteristics of the final product, both with respect to permeability and hysteresis loss, can be obtained with a comparatively inexpensive open anneal at relatively low temperature as the final heat treatment or treatments, is another outstanding advantage of the process. It is true that superior results are obtainable with a final, high temperature box anneal, as will be evident from Example D. The exceedingly thin material must be supported during annealing or it may lose its flatness. It also must be separated to prevent sticking. Hence, the handling problem becomes a delicate one, involving added expense. Where the highest possible permeabilities are requisite, the added expense will be found justified; and doubtless handling techniques will be improved. But by my process very high permeabilities and low core losses can be obtained with inexpensive open anneals at low temperatures, whereas hitherto high permeabilities in exceedingly thin materials could not be obtained, irrespective of cost.

Modifications may be made in my process without departing from the spirit of my invention.

Having thus described my process in certain exemplary embodiments, what I claim as new and desire to secure by Letters Patent is:

1. A process of producing very thin silicon steel of substantially .5 to 7 mils in thickness, having low hysteresis loss and a high permeability in the rolling direction which comprises producing from silicon steel containing substantially 2% to 4.5% silicon a starting material with a thickness substantially between 7 and 25 mils, low in carbon, and having a [100] (110) type of crystal orientation, and thereafter reducing said silicon steel starting material by cold rolling it in at least one stage with a cold rolling reduction of substantially between 60 and 90%, following each such stage of the said cold rolling with a recrystallizing heat treatment at a temperature substantially between 1200 and 2200° F.

2. The process claimed in claim 1 wherein the starting material is a silicon steel containing substantially from 2.90 to 3.30 silicon and has a carbon content not greater than substantially .007% together with an average grain diameter of from substantially .05 to 15 mm.

3. The process claimed in claim 1 wherein the starting material is a silicon steel containing substantially 2.90 to 3.30% silicon, not more than substantially .007% carbon, substantially .06 to .12% manganese, the remainder being iron, with a total oxide content of not more than substantially .025%, the said starting material having a straight grain permeability at 10 oersteds not less than substantially 1650, and having an average grain diameter of substantially .05 to 15 mm.

4. A process of producing thin silicon steel substantially .5 to 5 mils in thickness with high straight grain permeability and low hysteresis loss which comprises forming from hot rolled silicon steel containing substantially 2.90 to 3.30% silicon, by correlated cold rolling and annealing, a starting material of substantially 7 to 25 mils in thickness, having reduced the carbon content of said silicon steel to a value not greater than substantially .01%, the said starting material being subjected to a box anneal at above 2000° F. in dry hydrogen, said starting material having an average grain diameter of substantially .05 to 15 mm. and a high degree of preferred orientation of the [100] (110) type, and subsequently reducing said starting material by cold rolling it in at least one stage of from 60 to 90% reduction to a gauge of substantially .5 to 7 mils, and following each such stage with a recrystallizing heat treatment in a non-oxidizing atmosphere at a temperature substantially between 1200° F. and 2200° F.

5. The process of claim 4 wherein the said recrystallizing heat treatment is an open anneal.

6. The process of claim 4 wherein the said recrystallizing heat treatment is a high temperature box anneal in hydrogen.

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The following references are of record in the file of this patent:

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