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METHOD OF HEAT TREATING SILICON STEEL

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This invention relates to the production of materials to be used for electrical applications and particularly relates to the production of oriented silicon steels.

The use of silicon-iron alloys, commonly referred to as silicon steels, for electrical applications is well established commercially. An outstanding advance in that art concerned the orientation of the crystals and grains in silicon steel to produce unusual magnetic properties essentially like those characteristic of single crystals. Such advances have enabled the development, for example, of lighter transformers for a given capacity, and of electrical equipment that is more economical to operate because the operating characterisitics of the oriented materials normally do not change through aging.

A significant difficulty in this art has been the inability to obtain consistently satisfactory material that may be used for these purposes. Indeed it is common practice in steel manufacturing to test a small sample of hot rolled silicon steel to determine whether it is amenable to orientation and if it is not, to dispose of the steel as ordinary hot rolled strip. I know of no rational explanation of the inability heretofore, to orient silicon steel of one batch and then the seemingly inconsistent success with a succeeding batch melted to the same melting aim and prepared by the same steps as the unsatisfactory batch.

It is, therefore, an object of the present invention to provide a silicon steel strip which may be used to produce oriented silicon steel materials for use in electrical applications.

It is a further object of the invention to provide a process for the production of oriented silicon steel having the preferred orientation (110)[001], that can be practiced with existing facilities; that does not require special training for its practice; by which the percentage of grains or crystals that are preferentially oriented per unit of steel is maximized; and that consistently results in a product of magnetic properties satisfactory for high grade electrical applications.

We are taught by metal science that the magnetic or mechanical properties of any metal or alloy are determined by composition and structure. It is further known that composition often influences structure so that they cannot be said to be mutually independent. Therefore, any process to improve an existing material must be designed in such a way that control of both composition and structure is obtained.

Considering the foregoing principles in the production of silicon steels having the preferred orientation (110)[001], I have been able to develop a process whereby the described orientation is maximized and the resulting oriented crystals are made to grow rapidly while the non-preferentially oriented crystals present do not change size materially. Thus, in accordance with my

discoveries, outstanding silicon steels characterized by the preferred orientation (110)[001] are produced by cold rolling a hot rolled silicon steel strip having a specified composition to bring about a reduction in thickness, whereupon the resulting strip is annealed at an elevated temperature after having been heated to that temperature at a very rapid rate. The annealed strip is again cold rolled to final gauge and again annealed, advantageously at a higher temperature than the first mentioned anneal while once again heating to the annealing temperature at a specified rapid rate. The strip may then be decarburized and given a high temperature anneal, heating to temperature at a relatively slow rate. The order of cold rolling, annealing, again cold rolling, followed by the second anneal must be carried out in the order just specified. However, the decarburizing step and the high temperature anneal, where used, may be alternated with respect to one another as commercial conditions dictate.

In this manner I have been able to produce silicon steels having the percentage of crystals with preferred orientation (110)[001] maximized, and to grow the thus preferentially oriented crystals essentially to the exclusion, or at least comparative exclusion, of growth of other crystals that are present. Of equal importance, these advantageous results are entirely reproducible and are not the hit-or-miss affair heretofore common in the art. The materials thus produced, as will be obvious, can be utilized in the same manner that oriented silicon steels have been used heretofore, albeit with the increased efficiencies and other advantages that attend the particular characteristics of this material that have not been known generally heretofore.

The necessity of cold rolling, annealing, again cold rolling to gauge, followed by a second anneal in order to develop an oriented silicon steel has been known and has been disclosed in a number of patents. However, and in contradistinction to prior art, in the present invention it is essential that certain procedural limitations with respect to each of these steps be rigorously controlled if the advantages of this invention are to be obtained. The first cold rolling operation may be conducted in any manner desired so long as the reduction taken is within the range of about 60 to 80 percent, and preferably 70 to 80 percent. The second cold rolling likewise may be conducted with any apparatus and at any temperature utilized heretofore. However, the reduction taken in this cold roll must be in the range of about 50 to 60 percent of the then thickness of the strip. Though cold rolling, by definition, may be conducted at any temperature below the recrystallization temperature, for convenience I have found room temperature cold rolling to be particularly suitable.

The first anneal is a recrystallization anneal and, of course, it is essential that it be conducted above the recrystallization temperature for the material being processed. Suitably a temperature above about 850° C. is used for steels useful in this invention. The upper temperature limit is established by the necessity of keeping the size of crystals small at this time; an upper limit of about 1050° C. is effective for that purpose. The anneal, which is conducted in an inert or reducing atmosphere, is brought about by heating to the annealing temperature at a rate of about 1600° C. to 2000° C. per minute, e.g. about 1800° C. per minute. Such a heating rate is readily obtained by strand annealing or induction heating.

Orientation is a temperature-dependent variable and, as the preferred orientation is brought about above the

recrystallization temperature while other and undesirable characteristics result upon annealing at lower temperature, it is critically essential in the production of preferentially oriented material in accordance with my invention to heat to the annealing temperature so rapidly that appreciable softening does not occur while heating to temperature. On the other hand, crystal growth is a function of both temperature and time at temperature. To obtain the advantages of my invention, I have found it necessary to avoid undue crystal growth during this intermediate or recrystallization anneal; consequently, the annealing temperature must be held for a time sufficient to effect recrystallization but which does not exceed one minute, for it has been found that the desired recrystallization will occur within that time limit and at the temperature specified without incurring detrimental growth.

After the recrystallization annealing has been accomplished, the strip is again cold rolled, a reduction of 50 to 60 percent is imparted, after which the strip is again annealed in an inert or reducing atmosphere. The temperature conditions of the second anneal are similar to those of the first or recrystallization anneal above described, that is, a temperature of at least 850° C. to as high as about 1050° C. is used and is obtained by utilizing a heating rate of about 1600° C. to 2000° C. per minute. To avoid undesirable growth of crystals yet maximize the growth of those crystals having the preferred orientation, it is critically essential that the annealing temperature be attained rapidly to get to the temperaure at which the preferred crystals grow and to be at the intermediate temperatures, where the other crystals grow, a minimum time. It is for this reason that the above-mentioned heating rate is used in this second anneal. The material is held at the resulting annealing temperature for a time sufficient to completely soften the material being processed but not in excess of about one minute.

In each of the anneals described above the silicon steel was brought to the annealing temperature at a very rapid rate. Moreover, the annealing temperature was held for the short period of time not exceeding about one minute. While there are other features of the invention, as is apparent from this disclosure, it is my belief that the described heating rate and holding time are responsible for the superior characteristics of the materials produced in this invention.

After the second anneal the strip may be decarburized, for example, by heating it in an atmosphere of wet hydrogen at a temperature of about 600° C. to 800° C., and then given a final anneal at a temperature up to about 1000° C. to 1300° C. while heating to that temperature at a relatively slow rate, for example, on the order of 2° C. to 15° C., e.g. 5° C., per minute. If desired, this final anneal may precede the decarburizing step. In this event the strip is first cooled to below the recrystallization temperature and then is heated to the annealing temperature just described and is decarburized after annealing. The entire time of treatment which occurs above the recrystallization temperature in this final anneal should be at least sufficient for growth of the preferentially oriented crystals to be adequate, i.e. for at least one hour, yet should not be greater than about eight hours. A suitable procedure that may be used after the second cold roll involves passing the strip through a strand annealing furnace and coiling it as it emerges whereupon the resultant coil is decarburized and then box annealed slowly.

Silicon steels to which the instant process may be applied are in the form of hot rolled strip having a thickness on the order of 0.075 to 0.120 inch, preferably hot rolled and coiled at a temperature of not below about 800° C. It is desirable that such hot rolled silicon steel have a grain size of 1 mm. to 3 mm. To insure such a grain size, I have found it advantageous to give the hot rolled product as received an anneal at a temperature of about 1050° C. to 1150° C., prior to any cold working, for a time sufficient to secure that grain size.

The composition of the silicon steels that are useful contain, by weight, about 2.2 to 5.25 percent silicon, 0.001 to 0.05 percent of material selected from the group consisting of metal oxides, metal sulfides, and metal nitrides, a maximum of about 0.02 percent carbon, and the balance iron and incidental materials that do not deleteriously affect the desired results.

While I do not desire to have my invention limited by theory, it is my belief that to secure growth of preferentially oriented crystals while minimizing the growth of the other crystals present, it is necessary that a metal sulfide, nitride, or oxide in the defined amounts be present in the steel being treated. Indeed, the presence of that material in the specified amount, yet not exceeding 0.05 percent by weight, is critically essential to get reproducible results. Such impurity materials, I believe, must be those which can either be decomposed, be reduced, or go into solid solution at the annealing temperature. Typical of such materials are copper sulfide, manganese sulfide, alumina, aluminum nitride, and silicon dioxide. Steels of this composition can be made according to any process now used in producing electrical steels, though it is preferred that after they are hot rolled to the desired gauge, they are coiled at a temperature not below about 800° C.

The invention will be described further in conjunction with the following specific example. It should be understood that the details disclosed are not to be construed as limiting the invention.

A hot rolled silicon steel 0.100 inch thick having a composition within the specified range is heated to 1100° C. and maintained at that temperature until the average grain size is within 1 mm. to 3 mm. The strip is then cold rolled at room temperature using a conventional rolling mill, to reduce the strip to a thickness of 0.030 inch. The reduced strip is passed through a strand annealing furnace, having a dry hydrogen atmosphere, maintained at a temperature of 950° C. at a rate sufficient to insure that the strip remains in the furnace for a period of one minute. The strip is then again cold rolled to a thickness of 0.014 inch, at room temperature, whereupon the resultant strip is again strand annealed in dry hydrogen at 950° C. for one minute or the time required just to soften the material. As the strand leaves the annealing furnace, it is coiled and is placed in a furnace through which wet hydrogen is circulated continuously. The temperature in the furnace is maintained below 800° C. After one-half hour in the decarburizing furnace, the coiled strip is again annealed in a box annealing furnace by heating to 1200° C. at a temperature increase of 5° C. per minute. The atmosphere during this latter anneal is maintained inert and the anneal is continued for four hours. Subsequent tests of this material as determined by X-ray analysis, torque curves, magnetic character, and crystallographic studies show the steel to have the preferred orientation (110) [001], and that the crystals so oriented extend essentially throughout the material.

In other tests silicon steels produced in accordance with this invention were obtained and tested. A sample having a final thickness of 0.012 inch and an analysis as follows:

	Percent
Si	2.92
C	0.0053
N	0.0007
O_2	0.012
Fe	Balance

had the following magnetic properties:

Coercive force	0.125
Maximum permeability	52,000
Residual induction from a tip induction of 15,000 gauss	12,200
Hysteresis loss in watts per lb. per cycle	0.0042

A sample of the same material was also tested for total watt loss per pound at 15,000 gauss at various frequencies. The data are:

W-60	.59
W-120	1.66
W-180	3.00
W-240	4.84
W-320	6.84
W-360	8.91

From these data, it will be readily apparent that materials produced in accordance with my process set forth above are unusually useful for various electrical applications.

In accordance with the provisions of the patent statutes, I have explained the principles of my invention and have described what I now believe to be its best embodiment. However, it should be understood that variations from the details disclosed may be made without departure from the spirit and scope of my invention.

I claim as my invention:

1. In a method of producing oriented silicon steel, the step which comprises cold rolling a hot-rolled silicon steel strip of a thickness of about 0.075 to 0.120 inch having a composition consisting essentially, by weight, of about 2.2 to 5.25 percent of silicon, 0.001 to 0.05 percent of at least one material of the group consisting of metal oxides, metal sulfides and metal nitrides, a maximum of about 0.02 percent of carbon, and the remainder iron, to reduce said strip about 60 to 80 percent, heating said cold rolled strip at a rate of about 1600° C. to 2000° C. per minute to a temperature of about 850° C. to 1050° C. and maintaining said strip at said temperature for a time sufficient to effect recrystallization, cold rolling the thus heat-treated strip to effect a further reduction of about 50 to 60 percent, based on the then thickness, and again heating said strip at a rate of about 1600° C. to 2000° C. per minute to a temperature of at least about 850° C. and maintaining said temperature for a period of time just sufficient to soften the material, then subjecting the strip to a high temperature anneal for a period of from 1 to 8 hours at a temperature of from 1000° C. to 1300° C., said high temperature being attained by heating at a rate of from 2° C. to 15° C. per minute, each of said anneals being conducted in a non-oxidizing atmosphere and subjecting the strip to a decarburizing anneal at a temperature not exceeding 800° C. in wet hydrogen, the order of precedence of the high temperature final anneal and the decarburization anneal being immaterial.

2. A method according to claim 1 in which said recrystallization anneal is effected by strand annealing said strip for a period not in excess of one minute.

3. A method according to claim 1 in which said hot rolled silicon steel strip is annealed at a temperature of about 1050° C. to 1150° C. for a period of time sufficient to have the average grain size within about 1 mm. to 3 mm. prior to said first cold working operation.

4. A method of producing oriented silicon steel which comprises cold rolling a hot-rolled silicon steel strip of a thickness of about 0.075 to 0.120 inch having a composition consisting essentially, by weight, of about 2.2 to 5.25 percent of silicon, 0.001 to 0.05 percent at least one material of the group consisting of metal oxides, metal sulfides, and metal nitrides, a maximum of about 0.02 percent of carbon, and the remainder iron, to reduce said strip about 60 to 80 percent, heating said cold rolled strip at a rate of about 1600° to 2000° C. per minute to a temperature of about 850° to 1050° C. and maintaining said strip at said temperature for a time sufficient to effect recrystallization but not in excess of about one minute, cold rolling the thus recrystallized strip to effect a further reduction of about 50 to 60 percent, based on the then thickness, again heating said strip at a rate of about 1600° C. to 2000° C. per minute to a temperature of at least about 850° C. and maintaining said temperature for a period of time to just soften the sheets, then decarburizing said strip at a temperature below about 800° C., and high-temperature annealing it at a temperature of about 1000° C. to 1300° C. said high temperature annealing temperature being attained by heating to temperature at a rate of 2° C. to 15° C. per minute, each of said anneals being conducted in a non-oxidizing atmosphere.

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