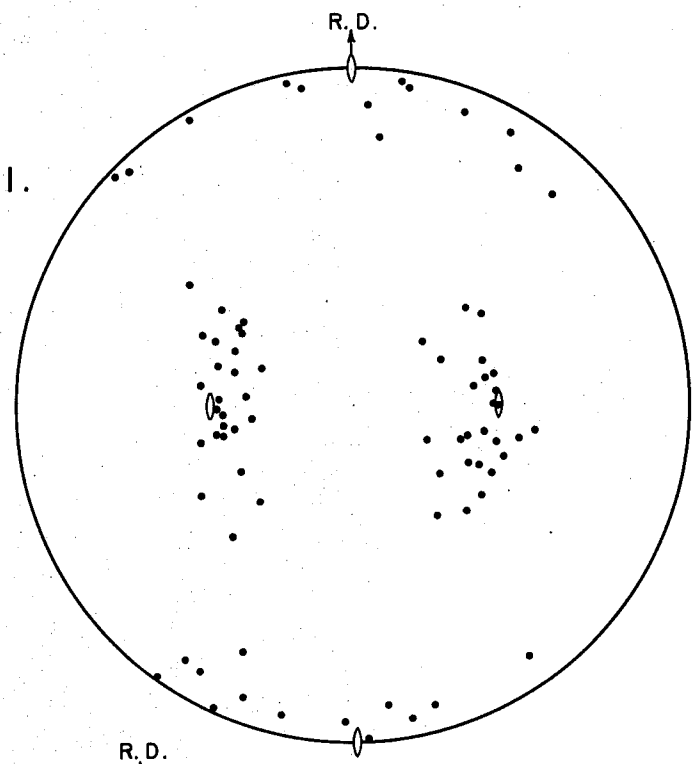
Fig. 1.



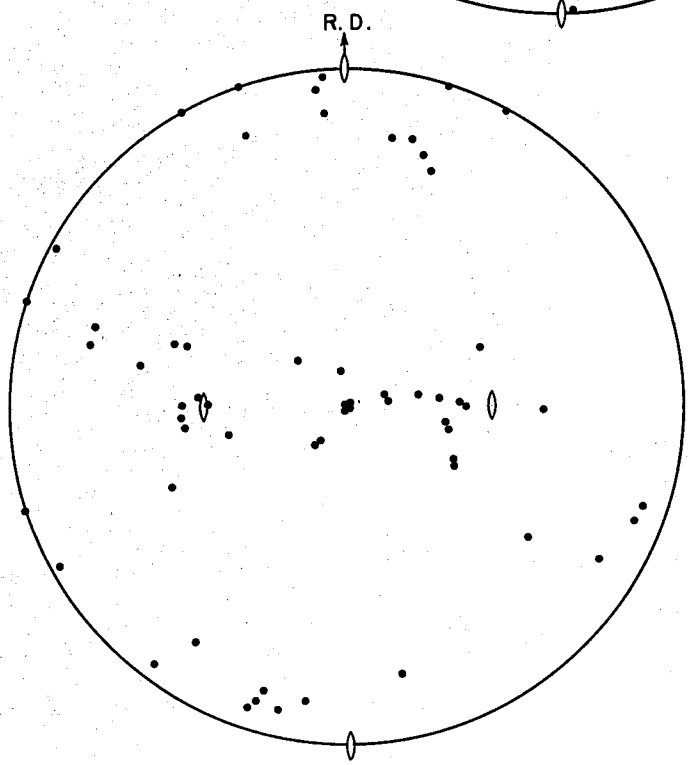


Fig. 2.

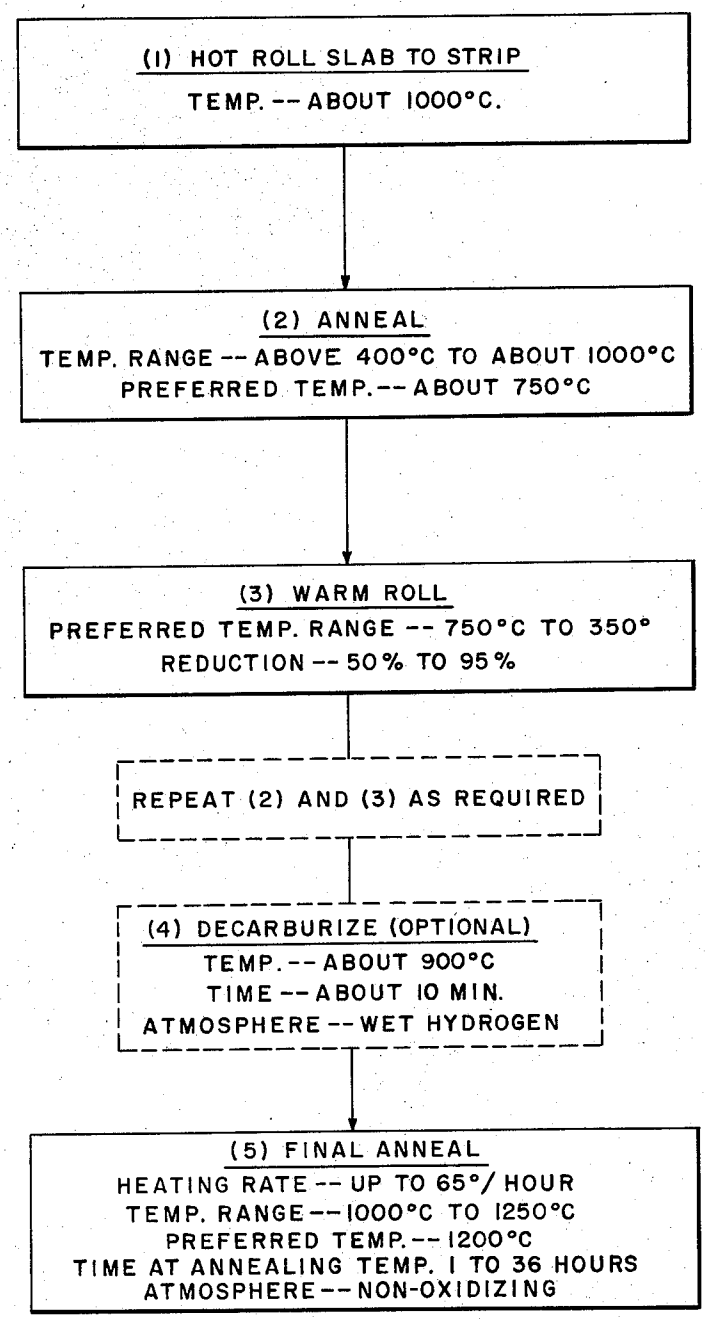

(1) HOT ROLL SLAB TO STRIP
TEMP. -- ABOUT 1000°C.
(2) ANNEAL
TEMP. RANGE -- ABOVE 400°C TO ABOUT 1000°C
PREFERRED TEMP. -- ABOUT 750°C
(3) WARM ROLL
PREFERRED TEMP. RANGE -- 750°C TO 350°
REDUCTION -- 50% TO 95%
REPEAT (2) AND (3) AS REQUIRED
(4) DECARBURIZE (OPTIONAL)
TEMP. -- ABOUT 900°C
TIME -- ABOUT 10 MIN.
ATMOSPHERE -- WET HYDROGEN
(5) FINAL ANNEAL
HEATING RATE -- UP TO 65°/ HOUR
TEMP. RANGE -- 1000°C TO 1250°C
PREFERRED TEMP. -- 1200°C
TIME AT ANNEALING TEMP. 1 TO 36 HOURS
ATMOSPHERE -- NON-OXIDIZING

Fig. 3.

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3,144,363

PROCESS FOR PRODUCING ORIENTED SILICON STEEL AND THE PRODUCT THEREOF

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This invention is directed to the manufacture of silicon steel for electrical purposes.

More particularly, the electrical steel produced by this invention is a high silicon alloy having an extremely low magnetostriction. Further, electrical steel made in accordance with this invention has a near "cube-on-edge" orientation.

Electrical steel sheet containing about 3% silicon and having "cube-on-edge" orientation, or as it is more conventionally identified in terms of Miller indices, the "(110)[001]" texture, has attained great commercial importance. Single oriented magnetic sheets of the type described and magnetic cores made therefrom are often sold under the trade name "Hipersil."

Transformers having laminations made from this low silicon "cube-on-edge" steel perform very satisfactorily in most instances. However, transformers of this type do produce a substantial amount of noise during operation, which noise is the result of the phenomenon known as magnetostriction. Many materials when magnetized or demagnetized undergo changes in dimension, the effect called magnetostriction, which is defined as:

$$\frac{\Delta L}{L}$$

or the change in length per unit length in the direction of magnetization under the magnetizing force applied It will be understood that the reduction of audible noise produced by transformers is an end much sought since there is currently a trend toward locating power substations in or near residential areas, or in office buildings and in shop areas.

As magnetic materials have improved with respect to core loss and permeability, it has become feasible to operate transformers at higher inductions than have been utilized heretofore. The magnitude of the magnetostrictive effect, however, and the noise resulting therefrom, increases with the induction, and for this reason, in many cases, it has been necessary to operate transformers below the maximum induction at which they are capable of operating.

There have been numerous suggestions for noise reduction in power transformers, some of which are shown in U.S. Patent No. 2,776,020, issued January 1, 1957, U.S. Patent No. 2,731,606, issued January 17, 1956, and in U.S. Patent No. 2,494,343, issued January 10, 1950.

Another suggestion for the reduction of noise in induction apparatus has been the use of unoriented steel having a relatively high silicon content and inherently low magnetostriction, for example, a steel containing about 6% silicon, but this has in the past proved impractical due to the extreme brittleness of such core material.

It is, therefore, a primary object of the present invention to reduce noise in induction apparatus such as transformers caused by magnetostrictive effects by employing a high silicon core steel initially containing critical amounts of sulfur and manganese which has been carefully heat treated and worked in a ductile condition to produce a near "cube-on-edge" texture. By "near cube-on-edge" texture it is meant that perfection of orientation is not attained and the texture is characterized by some scatter centering about the (110)[001] orientation.

It is a further object of the invention to reduce the noise level of transformer cores by employing a high silicon oriented core steel which has been carefully treated to obtain secondary recrystallization of near (110)[001] grains by a process which includes a final anneal in which the temperature of the alloy sheet is raised to final annealing temperature at a critically slow rate.

Other objects of the invention will, in part, be obvious and will, in part, appear hereinafter.

For a better understanding of the nature and objects of the invention, reference should be had to the following detailed description and to the drawings, in which:

FIGURE 1 is a (100) pole figure obtained on the alloy sheet after processing in accordance with this invention; and,

FIGURE 2 is a (100) pole figure obtained on alloy sheet which did not develop the desired texture; and,

FIGURE 3 is a flow diagram of the working schedule and heat treatment of the invention showing the conditions under which the various steps of the process are performed.

It has been found that extremely low magnetostriction and hence a low noise level may be obtained in sheets of an alloy initially composed of, by weight, from 4.5% to 7% silicon, from 0.10% to 0.50% manganese, from 0.01% to 0.10% sulfur, and the balance essentially iron except for a small amount of incidental impurities, when it is formed and carefully heat treated in accordance with the following process: (1) hot rolling slabs of the alloy material to strip, (2) heating the strip to an annealing temperature above 400° C., and preferably of about 750° C. in a reducing atmosphere, (3) warm rolling the heated strip at least once to effect a reduction in thickness of from 50% to 95%, (4) decarburizing, and effecting final anneal by heating the sheet to increase the temperature gradually at a rate of up to 65° C. per hour to a final annealing temperature of from 1000° C. to 1250° C. and holding at the final annealing temperature for a period of from 1 to 36 hours in a reducing atmosphere to complete secondary grain growth and remove impurities such as carbon and sulfur to a low level. A flow diagram of this process is set forth in FIG. 3.

While in the above process only a single stage of warm rolling and annealing has been described, in some cases it is desirable to employ two such stages, and even more stages would be within the scope of this invention, since the number of stages will depend on the initial thickness of the strip. The annealing temperature prior to warm rolling is stated to be above 400° C. and preferably about 750° C., but it should be understood that the upper limit for this anenal is about 1000° C. Sheets having a thickness in the range of 1 to 30 mils may be made by this warm rolling procedure.

In some cases, annealing and warm rolling may be used to produce strip having a thickness of about 10 mils, at which thickness the material becomes more ductile, and thereafter cold rolling may be employed to reduce the sheet to final gauge; the cold rolling being performed on sheet in the warm rolled condition.

Since carbon has a deleterious effect on magnetic properties and on grain boundary mobility, the decarburization anneal prior to the final anneal set forth in the above process, is desirable although not mandatory. An example of this treatment consists of an anneal for about 10 minutes at about 900° C. in wet hydrogen. Suitable wet hydrogen is hydrogen having a dew point of about 20° C. to 25° C.

The slow rate of heating to the final annealing temperature is critically necessary to obtain the desired secondary recrystallization texture.

The furnace atmosphere employed in the final annealing is hydrogen having a dew point of less than −25° C. or a vacuum of less than 10^{-3} mm. Hg. The principal requirement for the furnace atmosphere is that it be non-oxidizing.

In the alloys of this invention containing from 4.5% to 7% silicon, the relatively large amounts of manganese and sulfur required in the alloys have the function of inhibiting normal grain growth and promoting the growth of (110) secondaries by secondary recrystallization. The following example illustrates the great importance of manganese and sulfur concentrations in these alloys and the criticality of slow heating to the final annealing temperature upon the growth of (110) secondary crystals.

EXAMPLE

The compositions of typical heats are given below in Table I.

Table I

Heat No.	Silicon	Manganese	Sulfur	Phosphor	Carbon	Copper
MD-108	6.49	0.16	0.022	0.0003	0.028	0.02
MD-105	6.72	0.16	0.007	0.0001	0.012	

Both heats were made by air induction melting and were 500 to 1000 pounds in weight. Heat MD–108 (higher sulfur) was made with Armco iron whereas heat MD–105 was made with electrolytic iron of higher purity. Other additions were a commercial grade of silicon, electrolytic manganese and a small quantity of calcium-silicon for deoxidation.

These melts were poured into square ingot molds. One hour after pouring, each ingot was stripped hot and quickly placed in a furnace at 700 to 800° C. This step has been found to be desirable since thermal cracks may form if the material is allowed to cool to room temperature at this stage of the process. Subsequently, each ingot was heated to 1000° C. and held at 1000° C. for three hours.

Next, each ingot was initially forged at 1000° C. to a slab of a thickness of three inches. The material was reheated to 1000° C. when necessary during forging. Upon the completion of forging, the slab at about 700° C. was placed in a furnace at 700° C. and heated to 1000° C. After holding the slab at 1000° C. for one hour, the slab was hot rolled from a thickness of three inches to a strip of 0.14 inch and allowed to cool to room temperature. Slabs of thicknesses of as low as one inch may be forged or hot rolled, and then hot rolled into strip of suitable thickness, for instance from 50 mils to 200 mils.

The hot rolled steel strip was placed between mild steel plates to form a pack and the pack was then heated to and held at 750° C. for one hour. Then the material was pack rolled to a final thickness of about 20 mils at temperatures varying from 750 to 350° C. as the pack cooled.

Sections of the warm rolled strips from each heat were annealed at 1200° C. in dry hydrogen for periods of time of a few minutes to 16 hours. Two heating rates were used: slow heating at approximately 50° C./hour and fast heating obtained by inserting the samples in a furnace at 1200° C.

Upon inspection of the sheets treated in accordance with the above example, it is noted that secondary grains and particularly grains with diameters greater than five times the sheet thickness of the (110) type grew only in heat MD–108 (the high sulfur heat) when heated to final annealing temperature at the slow rate. Heat MD–105 did not undergo secondary recrystallization during either of the anneals. This indicates the necessity for sufficient quantities of sulfur in the sheets. Further, rapid heating of MD–108 at 1200° C. failed to grow (110) secondaries, and this is due to the fact that sulfur was removed too quickly at 1200° C. and therefore sufficient time was not available for the (110) grain nuclei to form and to grow in a fine grain matrix (that is, grains with diameters less than sheet thickness).

The (100) pole figure of FIG. 1 shows the texture of secondaries in MD–108 after slow heating to 1200° C. in a vacuum of less than 10^{-3} mm. Hg is close to (110)[001]. The rolling direction in the figure is indicated by the abbreviation "R.D." The ideal orientation (110)[001] is represented on the equator and in the rolling direction of the pole figure by the symbol (). The clustering of the poles about the (110)[001] position as shown in FIG. 1 was not obtained in any of the other sheets in which secondary recrystallization did not occur, but instead, there was obtained a nearly random distribution of planes of the (100) pole figure obtained from a sheet in which secondary recrystallization did not occur.

The magnetostriction of certain alloy sheets having the (110)[001] secondary recrystallization texture made by this process amounted to only 0.02 microinch per inch at 60 cycles per second at an induction of 10 kilogauss. This represents a very substantial improvement over the single oriented low silicon electrical steel sheet currently in use. A typical value for the magnetostriction of such low silicon steel under the same conditions is 1.0 microinch per inch. This is an improvement of the order of 50 times less movement.

In the alloys of this invention copper can replace some, and perhaps as much as 60%, of the manganese. Also, titanium and chromium may be substituted for part or all of the manganese. The purpose of the manganese, copper, sulfur, and other additives, as noted above, is to restrict the grain size of the matrix in which (110) grains grow and thereby increase growth rate of the secondary crystals. The reasons for the preferential growth of (110) grains in a fine-grained matrix containing growth inhibitors is not fully understood, but this seeming paradox has nevertheless been observed and utilized to produce large (110) grains in sheet steel.

Broadly, processing of the high silicon alloy containing substantial quantities of manganese and sulfur consists of hot rolling, at least one stage of annealing and warm rolling, and a final critical secondary recrystallization anneal. Each stage of annealing and warm rolling consists of an anneal above 400° C. and a warm reduction of 50% to 95% by rolling, the temperature during warm rolling being well above room temperature and sufficient to render the material ductile. In accordance with one successful process the secondary recrystallization anneal (the final anneal) may be effected by placing decarburized material in a furnace at about 750° C., heating at 60° C. per hour to 1100° C., holding 16 hours at 1100° C., heating at 25° C. per hour to 1200° C., and holding 2 hours at 1200° C. As a rough approximation the heating rate thus obtained is 17° C. per hour.

Annealing in a magnetic field has been found to improve the magnetic characteristics of these alloys, and therefore may be used where desired.

Since certain changes may be made without departing from the scope of the invention, it is intended that the accompanying description be interpreted as exemplary rather than limiting.

We claim as our invention:

1. In a process for producing sheets of oriented magnetic alloy having extremely low magneto-striction from hot-rolled alloy strip, the alloy strip composed of, by weight, from 4.5% to 7% silicon, from 0.10% to 0.50% manganese, from 0.01% to 0.10% sulfur, and the balance essentially iron except for small amounts of impurities, the steps comprising (1) heating the strip to a temperature of about 750° C., (2) warm rolling the strip in the temperature range of from 750° C. to 350° C. to effect a reduction of from 50% to 95%, (3) and finally annealing the sheets by slowly heating the sheet thus obtained at a rate of up to 65° C. per hour to a temperature of

from 1000° C. to 1250° C. in a non-oxidizing atmosphere and maintaining the sheet at this final annealing temperature to effect a high degree of secondary recrystallization and thereby produce a near (110)[001] grain orientation in the sheet.

2. The process of claim 1 in which the sheet is decarburized prior to final anneal by annealing the sheet at about 900° C. in wet hydrogen for about 10 minutes.

3. The process of claim 1 in which the final annealing temperature is about 1200° C.

4. In a process for producing sheets of oriented magnetic alloy having extremely low magneto-striction from a hot-rolled strip in which the alloy strip is composed of, by weight, from 4.5% to 7% silicon, from 0.10% to 0.50% manganese, from 0.01% to 0.10% sulfur, and the balance essentially iron except for small amounts of impurities, the steps comprising (1) annealing the strip at a temperature above 400° C., (2) warm rolling the strip to a reduction of from 50% to 95%, (3) repeating steps (1) and (2), (4) heating the sheet in a non-oxidizing atmosphere to increase the temperature at the rate of up to 65° C. per hour to a final annealing temperature of from 1000° C. to 1250° C. and maintaining the sheet at temperatures to obtain a near (110)[001] grain texture in the sheet by substantially complete secondary recrystallization.

5. The process of claim 4 in which the sheet is subjected to a decarburizing treatment prior to final anneal, the decarburizing treatment consisting of annealing the sheet at about 900° C. in wet hydrogen for about 10 minutes.

6. In a process for producing sheets of single oriented magnetic alloy having extremely low magneto-striction from an alloy slab composed of, by weight, from 4.5% to 7% silicon, from 0.10% to 0.50% manganese, from 0.01% to 0.10% sulfur, and the balance iron except for small amounts of impurities, the steps comprising (1) hot rolling the slab to strip at a temperature of about 1000°

C., (2) annealing the strip at a temperature above 400° C., (3) warm rolling the strip to a reduction of from 50% to 95% to produce sheet having a thickness of 1 to 30 mils, (4) decarburizing the sheet, (5) and finally annealing the sheet by slowly heating the sheet at a rate of up to 65° C. per hour to a temperature in the range of 1000° C. to 1250° C. in a non-oxidizing atmosphere and maintaining the sheet at this final annealing temperature for a period of time sufficient to effect a high degree of secondary recrystallization and thereby produce a near (110)[001] grain texture in the sheet.

7. In a process for producing sheets of single oriented magnetic alloy having extremely low magnetostriction from an alloy slab composed of, by weight, about 6.5% silicon, from 0.10% to 0.50% manganese, from 0.01% to 0.10% sulfur, and the balance iron except for small amounts of impurities, the steps comprising (1) hot rolling the slab to strip at a temperature of about 1000° C., (2) annealing the strip at a temperature of about 750° C. for about 1 hour, (3) warm rolling the strip at temperatures of from 750° C. to 350° C. to a reduction of from 50% to 95% to produce sheet having a thickness of 1 to 30 mils, (4) decarburizing the sheet, (5) and finally annealing the sheet by slowly heating the sheet at a rate of about 50° C. per hour to a temperature of about 1200° C. in dry hydrogen and maintaining the sheet at this final annealing temperature for a period of time sufficient to effect a high degree of secondary recrystallization and thereby produce a near (110)[001] grain texture in the sheet.

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