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PROCESS FOR PRODUCING CUBE-ON-EDGE ORIENTED SILICON IRON

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Fig. 1.

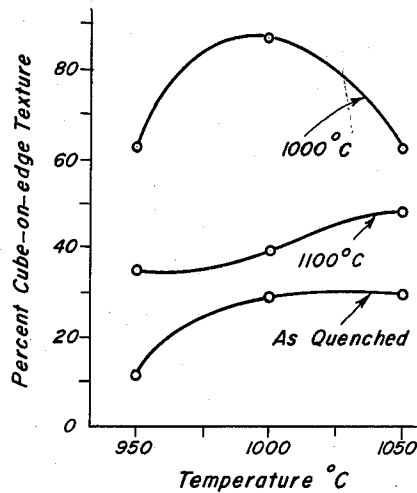
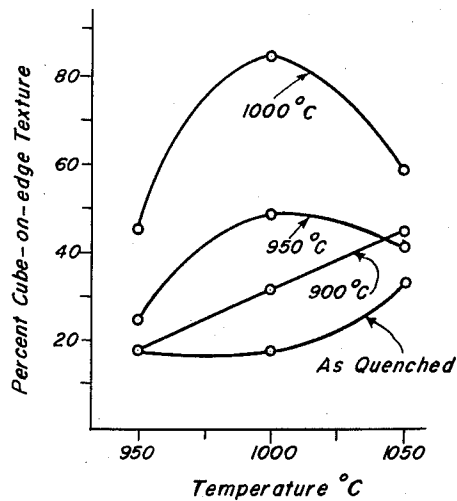


Fig. 2.



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PROCESS FOR PRODUCING CUBE-ON-EDGE
ORIENTED SILICON IRON

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This invention relates to the fabrication of polycrystalline, magnetically soft rolled sheet metal composed principally of an alloy of iron and silicon having a high percentage of the grains comprising the material oriented such that their crystal space lattices are arranged in a substantially identical relationship to the plane of the sheet and to a single direction in the plane of the sheet, and more particularly to an improved process for forming sheet material having this desired orientation.

The sheet materials to which this invention is directed are usually referred to in the art as "electrical" silicon steels or, more properly, silicon-iron and are conventionally composed principally of iron alloyed with about 1½ to 4 percent, preferably 2½ to 3½ percent silicon, and relatively minor amounts of various additions or impurities such as sulfur, manganese, vanadium, titanium, phosphorus and a very low carbon content as finished material. Such alloys crystallize in the body-centered cubic crystallographic system at room temperature. As is well known, this refers to the symmetrical distribution or arrangement which the atoms forming the individual crystals or grains assume in such materials. In these materials, the smallest prism possessing the full symmetry of the crystal is termed the unit cell and is cubic in form. This unit cube is composed of 9 atoms, each arranged at the corners of the unit cube with the remaining atom positioned at the geometric center of the cube. Each unit cell in a given grain or crystal in these materials is substantially identical in shape and orientation with every other unit cell comprising the grain.

The unit cells which are body-centered unit cubes comprising these materials each have a high degree of magnetic anisotropy with respect to the crystallographic planes and directions of the unit cube, and hence, each grain or crystal comprising a plurality of such unit cells exhibit a similar magnetic anisotropy. More particularly, crystals of the silicon-iron alloys to which this invention is directed are known to have their direction of easiest magnetization parallel to the unit cube edges, their next easiest direction of magnetization perpendicular to a plane passed through diagonally opposite parallel unit cube edges, and their least easiest direction of magnetization perpendicular to a plane passed through a pair of diagonally opposite atoms in a first unit cube face, the central atom and a single atom at the unit face which is parallel to the first face. As is well known, these crystallographic planes and directions are conventionally identified in terms of Miller indices, a more complete description of which may be found in "Structure of Metals," C. S. Barrett, McGraw-Hill Company, New York, New York, Second Edition, 1952, pages 1-25, and conventionally referred to as, respectively, the (100) plane and the corresponding [100] direction, the (100) plane and the [110] direction, and the (111) plane and the [111] direction.

It has been found that certain of the silicon-iron alloys may be fabricated by rolling and heat treatment to form sheet or strip material composed of a plurality of crystals or grains, a majority of which have their atoms arranged so that their crystallographic planes have a similar or substantially identical orientation to the plane of the sheet or strip in a single direction in said plane. This material is usually referred to as "oriented" or "grain oriented" silicon-iron sheet or strip and is characterized by having a majority of its constituent grains oriented so that four of

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the cube edges of the unit cells of said grains are substantially parallel to the plane of the sheet or the strip and to the direction in which it was rolled and a (110) crystallographic plane substantially parallel to the sheet. It will thus be seen that these so-oriented grains have a direction of easiest magnetization in the plane of the sheet in the rolling direction and the next easiest direction of magnetization in the plane of the sheet in the transverse rolling direction. This is conventionally referred to as a "cube-on-edge" orientation or the "(110)[001] texture." In these polycrystalline sheet and strip materials, it is desirable to have as high a degree of grain orientation as is attainable in order that the magnetic properties in the plane of the sheet in a rolling direction may approach the maximum attained in a single crystal in the (100) direction.

It has been recognized that the development of the cube-on-edge grain orientation in silicon-iron material is controlled by inclusions such as manganese sulfide, titanium carbide, vanadium carbide or vanadium nitride, and to a lesser extent, silicon nitride. These inclusions or second phase precipitates perform the function of restraining normal grain growth and thereby promoting the selective growth of the (110)[001] oriented grains during the final recrystallizing anneal. In the absence of inclusions, many grains of widely differing orientations grow and no sharp texture is obtained. Additionally, it has been shown that the driving energy for the growth of the cube-on-edge oriented grains is inversely proportional to the diameter of the matrix grains, so that the more effectively the inclusions maintain a small matrix grain size, the more readily the grains of the desired orientation grow. This type of grain growth behavior, where a few grains grow large at the expense of many small grains, is generally referred to as secondary recrystallization.

The effectiveness of inclusions in promoting secondary recrystallization is dependent upon their number, size, and distribution throughout the matrix silicon-iron alloy. I have shown that for a given weight content of inclusion forming metal-non-metal additions, the number, size and distribution of inclusions is determined by the rate of cooling from a temperature at which the inclusions are in solution. A slow rate of cooling through and below the solubility limit favors the formation of few inclusions and their subsequent growth, whereas more rapid cooling promotes the formation of many small inclusions. Also, a material with many small inclusions has greater resistance to normal grain growth and consequently develops a higher degree of cube-on-edge orientation in the final anneal than material with fewer and larger inclusions.

In actual steel mill practice, these silicon-iron alloys are prepared by casting large, thick section ingots weighing up to about 4 tons each from alloys containing from about 1½ to 4 percent, by weight, silicon. Additionally, in order to form a second phase dispersion, or the inclusions required for retarding normal grain growth, relatively minor amounts of metal and non-metal additions are included. Specifically manganese additions are normally made in amounts of up to about 0.15 percent, while the non-metallic additions are normally in amounts of up to about 0.035 percent carbon, about .03 percent sulfur, and up to about .005 percent nitrogen. Those special alloys in which vanadium or titanium are used will generally have these elements added in amounts of up to about 2.0 and 0.10 percent, respectively. By thick section, it will be understood that such ingots usually have a minimum transverse cross-sectional dimension of about 2 feet. Such ingots are conventionally hot worked into a strip or sheet-like configuration, usually less than 150 mils in thickness, commonly referred to as "hot rolled band." This band material is usually in an incompletely recrystallized form and may be annealed to effect com-

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plete recrystallization, if desired, but this is usually not done in conventional commercial practice.

The hot rolled band is then cold rolled with appropriate intermediate heat treatment to the finished sheet or strip thickness, usually involving at least a 40 percent reduction in thickness, and given a final or texture producing annealing treatment accompanied by a decarburizing treatment.

It is a principal object of this invention to provide an improved process for producing a finely divided, evenly dispersed second phase in silicon-iron alloys which assists development of the cube-on-edge orientation.

Other objects and advantages of this invention will be in part obvious and in part explained by reference to the accompanying specification and drawings.

FIG. 1 is a graph showing the percent cube-on-edge texture as a function of the final recrystallizing annealing temperature for samples processed according to this invention and

FIG. 2 is a graph similar to that of FIG. 1 in which the silicon-iron alloys have been given a modified form of processing.

Generally, the process of the present invention comprises providing a silicon-iron alloy which includes some minor amounts of metal and non-metal additions which are capable of forming a finely divided, evenly dispersed second phase precipitate throughout the silicon-iron alloy. The alloy is heated to a temperature above that at which the minor additions are in solution to preclude the precipitation of an effective second phase dispersion. Having dissolved the minor additions in the matrix alloy, the material is then quenched rapidly from above to below the solution temperature, but it is essential that this quenching be carried out at a rate sufficient to preclude the formation of the second phase particles, i.e., cooled rapidly enough that the additions remain in solution even though the body is at a temperature where precipitation would normally occur. Following this quenching, the bodies are then given an anneal which will effect precipitation of the minor additions in the form of an evenly dispersed, finely divided second phase precipitate.

Generally, the temperature for this annealing operation will fall within the range of from about 900–1100° C. although it will be appreciated that slight variations can be expected due to the differing natures of the additive materials which may be used. Once the second phase has been precipitated the material can then be cold rolled with intermediate anneals where necessary between rolling stages and then given a final recrystallizing anneal to effect secondary recrystallization with the consequent growth of the (110) [001] orientation from the otherwise fine, random matrix grains. As will be explained more fully later, the silicon-iron alloy can either be given the precipitation anneal immediately following quenching of the alloy from above the solution temperature or this treatment can be given following quenching of the body at the termination of hot rolling. In either event the annealing is done prior to any cold rolling.

To clearly illustrate the effect of the present invention on the obtainment of the cube-on-edge orientation, a charge of electrolytic iron, 98 percent ferrosilicon, iron sulfide, and manganese was induction melted under a cover of argon and poured at 1550° C. into a baked silica sand mold having a 3" x 5" cross section. The ingot contained 3.11 percent silicon, 0.022 percent sulfur, 0.056 percent manganese, 0.002 percent carbon, 0.003 percent oxygen, and 0.001 percent nitrogen.

A slice 3" x 5" x 1" thick was then taken from the ingot, heated in hydrogen to 1325° C., a temperature sufficiently high to insure the solution of the manganese sulfide, and then quenched in iced brine (about 5° C.). This quenched slab was then cut into three equal pieces, one of the pieces being heated to 900° C. and rolled without reheating to 0.10 inch thick band, a second piece being

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heated for 1½ hours at 1000° C. and then rolled from 900° C. to 0.10 inch thick band, and the third piece was heated for 1½ hours at 1100° C. before rolling from 900° C. to 0.10 inch band. All of the hot rolled bands were then pickled, heat treated for 5 minutes at 900° C., cold rolled to 0.025 inch, heat treated 2½ minutes at 860° C., and then rolled to a final gauge thickness of 0.012 inch. All of these specimens were studied both microscopically and macroscopically immediately following the brine quenching operation and following the precipitation producing heat treatments which were effected at temperatures of from 900–1100° C. It was found that the rapid quench of the 1 inch thick slab in the iced brine greatly restricted the formation and growth of manganese sulfide inclusions. Those inclusions visible at a magnification of 1000 diameters were primarily large oxide inclusions not of a nature to be considered effective for purposes of restricting normal grain growth during texture developing anneals. It was only after heating at 1000° C. that the second phase precipitate became clearly visible. When a temperature of 1100° C. was used the precipitate became much larger and hence not as effective in assisting production of cube-on-edge orientation as the particles produced by the 1000° C. anneal.

Pieces of the final gauge strip (0.012 inch) were heated for 2 hours in argon at 950, 1000 and 1050° C. The ultimate degree of texture developed in these materials can clearly be seen by reference to FIG. 1 of the drawings where the percent cube-on-edge texture developed at the various final annealing temperatures is shown as a function of the precipitation obtaining intermediate heat treatment. Specifically, it will be noted that optimum texture is obtained in the piece which was heat treated at 1000° C. following quenching in the iced brine. Additionally, while the 1100° C. heat treatment did develop a second phase precipitate, it is obvious that the amount of cube-on-edge texture obtained is vastly inferior to that obtained using the 1000° C. intermediate annealing temperature. It may be possible in some instances, of course, when the 1050° C. final annealing temperature is used that material produced or given the 1100° C. intermediate treatment will be useful.

The following discussion clearly indicates that the quenching can be effected either during or at termination of hot rolling so that the precipitation anneal can follow hot rolling without any deleterious effects on the size and distribution of the second phase and that in fact it may be possible to obtain properties slightly superior to those obtained when quenching preceded the hot rolling. Here, once again, a slice 3" x 5" x 1" thick was cut from the original ingot and heated to 1325° C. to place the manganese sulfide in solution. This body was then quickly reduced to 0.10 inch thick band by two passes through a reversing mill before quenching in iced brine. After pickling, a piece of the band was reduced to a final gauge of 0.012 inch by following the cold rolling, heat treating and cold rolling sequence described in connection with the previous samples. Other portions of the band were first heat treated for 1½ hours at 900, 950 and 1000° C. before similarly cold rolling to final gauge. The slab in this instance probably experienced a higher cooling rate in passing through and below the solubility limit than did the previous slab due to the difference in the thickness of the material when quenching was effected. A study of the microstructures produced in the bodies produced as just outlined indicated that no inclusions were visible in the as-rolled and quenched band. However, heating at 900° C. prior to cold rolling caused a heavy localized precipitation at such preferred sites as slip lines and grain boundaries but there was little general precipitation. Heating at 950° C. produced both general and localized precipitation while heating at 1000° C. produced essentially only general precipitation, the latter precipitation being that most advantageous to the restriction of nor-

mal grain growth and consequent growth of the (110) [001] oriented grains.

The degree of orientation developed in these four specimens can best be seen by referring to FIG. 2 of the drawings. Here it is obvious that the degree of ultimate cube-on-edge texture increases as the precipitation developing heat treatment temperature increases, optimum texture, of course, being obtained when both a 1000° C. precipitation anneal and a 1000° C. final anneal were used.

While the preceding examples all relate to the use of a manganese-sulfide second phase dispersion, it is obvious that the other precipitation forming minor ingredients mentioned earlier can just as effectively be used. Vanadium nitride is particularly useful since it can be readily removed following the recrystallizing anneal whereas the manganese sulfide requires a somewhat longer period of time.

Although the present invention has been described in connection with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and the appended claims.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. A process for producing silicon-iron bodies having a majority of the constituent grains oriented in the (110)[001] texture, comprising providing a silicon-iron body composed of from 1.5 to 4.0 percent silicon, balance substantially all iron and containing minor additions capable of forming a second phase dispersion, said additions being up to about 0.15 percent manganese, up to about 2.0 percent vanadium, up to about 0.10 percent titanium, up to about 0.035 percent carbon, up to about 0.03 percent sulfur and up to about 0.005 percent of nitrogen, quenching the silicon-iron alloy from a temperature not less than about 1325° C. where the minor additions are in solution at a rate retaining them in solution, heat treating the quenched alloy at a temperature of from about 900° C. to 1100° C. precipitating a finely divided,

evenly dispersed second phase assisting development of the (110)[001] grain orientation, cold rolling the quenched and heat treated alloy to a desired final thickness, and subjecting the cold rolled alloy to a final recrystallizing anneal at a temperature of from about 950° C. to 1050° C. to effect secondary recrystallization thereof and develop the desired (110)[001] crystalline orientation.

2. In the process for producing cube-on-edge grain oriented silicon-iron sheet having the steps of:

- (1) providing a starting body composed of from 1.5 to 4.0 percent silicon, balance substantially all iron and containing minor additions capable of forming a second phase dispersion, said additions being up to about 0.15 percent manganese, up to about 2.0 percent vanadium, up to about 0.10 percent titanium, up to about 0.035 percent carbon, up to about 0.03 percent sulfur and up to about 0.005 percent of nitrogen;
- (2) hot rolling the body to an intermediate thickness;
- (3) cold rolling the body to a final thickness, using intermediate anneals where necessary; and
- (4) effecting a final recrystallizing anneal developing the cube-on-edge orientation; the additional steps of:
- (5) quenching the body prior to cold rolling from a temperature of not less than about 1325° C. where the minor additions are in solution at a rate retaining them in solution; and
- (6) heat treating the quenched alloy at a temperature of from about 900 to 1100° C. precipitating a finely divided, evenly dispersed second phase assisting development of the cube-on-edge grain orientation.

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