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METHOD OF IMMERSING CUBE-ON-EDGE ORIENTED SILICON-IRON IN SILICON-IRON BATH FOLLOWED BY ANNEALING TO FORM A CUBE-ON-EDGE ORIENTED SILICON-IRON PRODUCT

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2 Claims. (Cl. 148—112)

This invention relates to polycrystalline soft magnetic sheet material and more particularly to silicon-iron alloy sheets containing 1.5 to 4.5 weight percent silicon, balance substantially all iron which have their constituent grains oriented in the (110) [001] crystallographic orientation.

This invention is a continuation of applicant's copending application Serial No. 199,285, filed June 1, 1962.

The sheet materials to which this invention is directed are usually referred to in the art as "electrical" silicon steels or, more properly, silicon irons and are conventionally composed principally of iron alloyed with about 1.5 to 4.5 percent and preferably about 2.5 to 3.5 percent silicon and relatively minor amounts of various impurities such as sulfur, manganese, phosphorous and having 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 nine 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 or 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 the unit cube, and hence, each grain or crystal comprising a plurality of such unit cells exhibits a similar 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 easy direction of magnetization perpendicular to a plane passed through 3 atoms situated at the end of 3 cube edges having a common vertex. 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 Book Company, New York, New York, second edition, 1952, pp. 1–25, and will be referred to as, respectively, the (100) plane and the corresponding [001] direction, the (110) plane and the [110] direction, and the (111) plane and the [111] direction.

It has been found that these silicon-iron alloys may be fabricated by unidirectional 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 and to 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 50 percent or more of its component grains oriented so that four of the cube edges of the unit cells of said grains are substantially parallel to the plane of the sheet or strip and to the direction in which it was rolled and a (110) crystallographic plane substantially parallel to the plane of 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 a rolling direction and the next easiest direction of magnetization in the plane of the sheet in the transverse to 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 and in the rolling direction may approach the maximum attained in single crystals in the [001] direction.

Prior processes for producing cube-on-edge oriented sheets, while effective, present many control problems which must be met if the orientation is to be obtained. For example, the presence of a finely-divided, evenly dispersed second phase should normally be present or good cube-on-edge orientation will not be obtained. Then, after the orientation is obtained, the dispersion must be removed if commercially magnetic properties are desired, this being a relatively costly and time consuming operation.

It is the principal object of this invention to provide an improved process for producing cube-on-edge oriented sheets of silicon-iron alloys.

An additional object of this invention is to provide a process for producing cube-on-edge oriented silicon-iron sheets which are produced by freezing molten alloys on oriented matrix sheets of about the same composition.

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

Generally, the process of this invention comprises providing a strip or sheet of a silicon-iron alloy containing the required amount of silicon, which strip or sheet has the cube-on-edge orientation, and then subjecting this strip to a cleansing anneal to remove any surface contaminants which may interfere with subsequent processing. The thus cleaned sheet is then immersed in a bath of molten silicon-iron alloy of substantially the same composition as the sheet to freeze additional metal thereon. The sheet thus performs the function of a matrix sheet, since it acts as a heat sink causing metal to freeze on it and further, by virtue of its orientation, causes nucleation in the newly frozen material such that it also becomes oriented in the (110) [001] crystallographic orientation. The matrix sheet and the metal accreted thereto is then air cooled and subsequently heat treated or annealed in a non-oxidizing atmosphere such as dry hydrogen.

Considering the process in more detail, the first step is that of providing a cube-on-edge oriented sheet which will act as a matrix upon which metal will be frozen when the sheet is immersed in a molten bath. It is not essential that the orientation present in the matrix sheet be derived by any particular process, it being important only that the sheet have the cube-on-edge orientation. Generally, the sheet will be relatively thin, i.e., no greater than about 25 mils, since the thickness of the matrix is appreciably increased by immersion. Preferably, the thickness of the matrix and the accreted metal should be kept below that at which hot rolling would become desirable, that is, maintained at thicknesses where cold rolling is adequate to reduce the product to final thicknesses up to about 15 mils.

Since the matrix acts to nucleate the growth of cube-on-

edge grains in the metal being frozen on it, it is essential that the surface be clean and free of any oxide film which would impede the epitaxial growth of the new grains. For this reason, the matrix sheet is initially subjected to a cleansing anneal in pure dry hydrogen. Generally, hydrogen having a dew point no higher than —67° F. will adequately cleanse the surface of the matrix sheet so that nucleation and growth can occur unimpeded

In view of the fact that the matrix sheet is comparatively thin, it is obvious that the immersion time in the molten silicon-iron alloy must be kept very small and that the temperaure of the bath itself preferably held at a temperature just above the liquidus. Immersion times of up to about 0.5 second for any given area of matrix surface with a bath temperature of about 1490° C. has proven effective. After immersion, the product is air cooled and then given a final anneal in a non-oxidizing atmosphere at temperatures ranging from about 1000 to 1350° C. If desired, this final anneal can be used to remove any impurities from the product which would effect adversely its magnetic properties. Should this be the case, an atmosphere of dry hydrogen (i.e. dew point —70° F.) will prove highly advantageous.

Considering a specific example of the present invention, a strip of 3¼ percent silicon-iron sheet 1 3/16 inches wide and 14 mils thick was annealed in dry hydrogen (—70° F.) at 850° C. for 60 minutes to cleanse the surface. The sheet was then immersed for ⅓ second in a silicon-iron bath at 1490° C., this temperature being just above the liquidus for this particular alloy. The product to input weight ratio of the sheet was 7 to 1 which was well above the theoretical ratio of 4 to 1. The product was then air cooled and given a final anneal in dry hydrogen (—70° F) at 1100° C. The final product was sectioned and the microstructure studied and it was found that the additional metal frozen on the matrix sheet had assumed the cube-on-edge orientation of the matrix.

It is thus apparent that this process provides a new and simplified method for producing cube-on-edge oriented silicon-iron. Once the product of this invention has been achieved, it can be rolled to thinner gauges by known techniques with no deterioration of the orientation.

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 sheets consisting essentially of from about 1.5 to 4.5 weight percent silicon, balance substantially all iron and having their constituent grains oriented in the (110) [001] crystallographic orientation, comprising the steps of subjecting a (110) [001] grain oriented matrix sheet of up to about 25 mils thickness to a dry hydrogen atomsphere having a dew point no higher than about —60° F. at a temperature of from about 700 to 1000° C. to clean the surface thereof, immersing the matrix sheet in a bath of molten silicon-iron alloy of substantially the same composition as the matrix to freeze additional metal thereon, air cooling the matrix sheet and frozen metal, and annealing the cooled product in a non-oxidizing atmosphere at temperatures of from about 1000 to 1350° C., the additional frozen metal having a (110) [001] crsytallographic orientation at the termination of processing.

2. A process as defined in claim 1 wherein the final anneal is carried out in hydrogen having a dew point no higher than about —70° F.

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