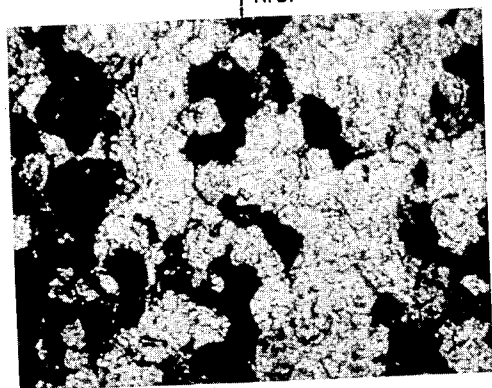
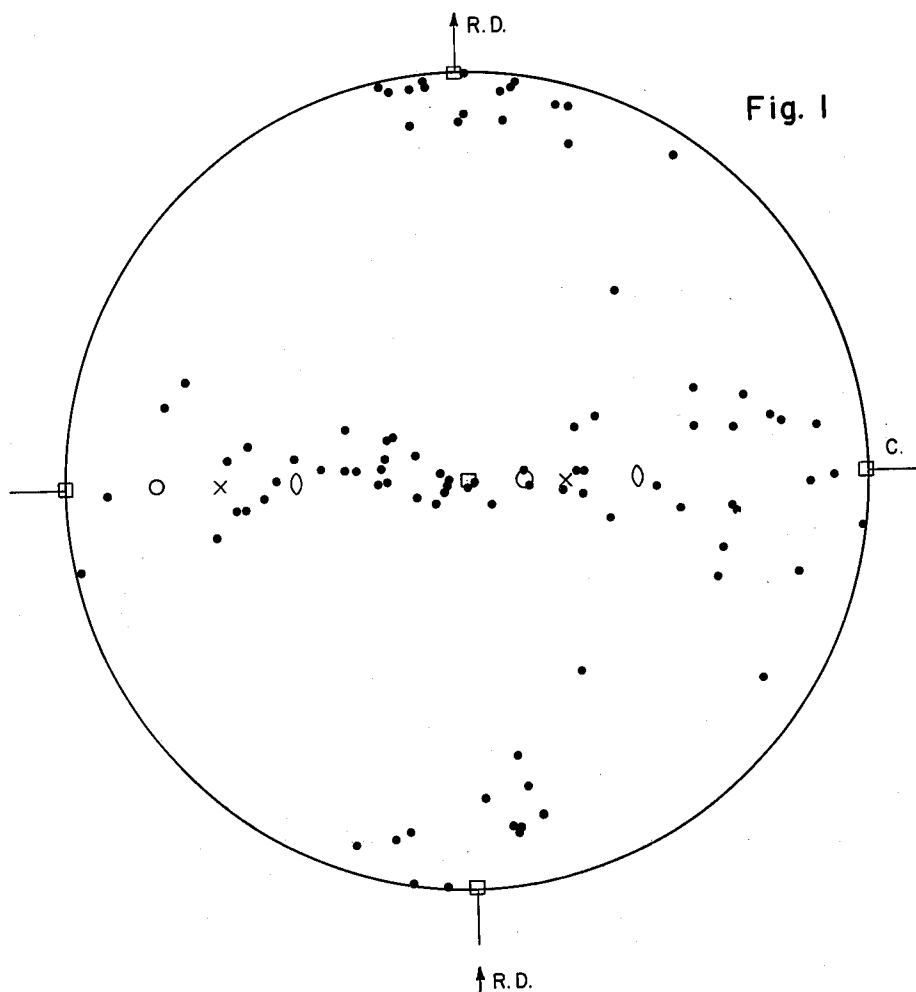


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METHOD FOR PRODUCING FIBER TEXTURE AND CUBE-TEXTURE
SHEETS OF IRON-BASE ALLOYS
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3,089,795

METHOD FOR PRODUCING FIBER TEXTURE AND CUBE-TEXTURE SHEETS OF IRON-BASE ALLOYS

Hsun Hu, Pittsburgh, Pa., assignor to Westinghouse Electric Corporation, East Pittsburgh, Pa., a corporation of Pennsylvania

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This invention relates to the field of soft magnetic materials and is particularly directed to a process for making polycrystalline iron-base sheet material having a highly developed fiber texture which may be further processed to produce cube-texture material.

The soft magnetic materials such as iron, and alloys of iron and silicon, iron and aluminum, and iron and molybdenum, have found wide industrial application as core materials in power transformers, motors, generators, and other applications. These cores are usually laminated structures made by stacking relatively thin sheets of magnetic materials which have been punched or sheared to shape or by winding a long strip into a closed loop structure, to form a core of the desired configuration. In general, but not exclusively, these laminated cores provide one or more closed magnetic circuits.

Because of the anisotropic character of the magnetic properties of single or individual crystals of the iron-base alloys of concern here, much work has been done to obtain preferred orientation of the crystals in polycrystalline material. The iron and iron base materials have a body centered cubic crystalline structure at low temperatures and retain the structure over a temperature range which extends up to several hundred degrees C.; for example, up to 910° C. for pure iron. The body centered cubic materials all have their direction of easiest magnetization in a direction parallel to one edge of the unit crystal cube.

A sheet of material having a large proportion of the grains thereof arranged to provide optimum magnetic properties in several directions may be described as having the orientation (100) [001]. The (100) notation denotes that a (100) crystallographic plane or cube face is in or parallel to the plane of the sheet, and the [001] notation denotes that a [001] crystallographic direction or four cube edges are parallel to the rolling direction of the sheet.

An oriented magnetic sheet material having somewhat less desirable magnetic properties, but nevertheless a material finding wide commercial application, is one wherein a majority of the grains of the material have an orientation which may be described as (110) [001]. This preferred orientation is commonly referred to as "cube-on-edge" texture since four cube edges are parallel to the rolling direction, but four of the cube faces are, on the average, at an angle of 45° to the sheet surface. Sheet material of this type is commonly produced by secondary recrystallization techniques. The "cube-on-edge" material in sheet form has magnetic properties which are very superior including a maximum magnetic induction (flux density) for a given magnetizing force and a maximum possible induction in the given material in the direction of rolling. However, in the direction transverse with respect to the direction of rolling these magnetic properties are relatively poor. This means that when the magnetic flux path coincides with the rolling direction good magnetic properties are obtained, but when the flux path changes to a direction transverse to the rolling direction, as, for example at a 90° corner in an L- or U-shaped punching, this portion has relatively poor magnetic properties with a corresponding reduction of the efficiency of the core.

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It has long been recognized for the above and other reasons that a polycrystalline sheet material of iron-base alloys having the optimum magnetic properties, substantially equal in the longitudinal and transverse directions and comparable to those previously available only in the rolling direction (for cube-on-edge material), would be of considerable utility in the field of electrical power apparatus. For example, circular magnetic punchings for motors and generators would be greatly improved if such magnetic sheets are available.

A practical process for the development of cube texture in iron-base alloys has eluded the efforts of numerous investigators. Most of the processes which have been disclosed to effect this desired end employ secondary recrystallization phenomena. It is a typical characteristic of a secondary recrystallization process that the amount of cube grains in the primary recrystallization structure is very small. In many instances, these primary cube grains consists of only a few percent of the total volume, and occasionally may reach 10 to 20 percent of the volume. Since the driving force for subsequent growth of these cube grains during secondary recrystallization depends upon the small differences in surface energy of various crystallographic faces, critical control of the furnace atmosphere, or the vacuum if that is used, is required. Even when annealing conditions are carefully controlled, growth of these cube grains may completely stop due to difficulties, the nature of which is not fully understood. Once growth of the cube grains has stopped, further annealing has little effect in restoring their ability to grow. On the contrary, prolonging the annealing treatment often causes the (110) grains to grow at the expense of the cube grains. It is known that this last difficulty can be overcome by repeatedly cleaning, by which is meant etching or electrolytic polishing, the surface of the specimen between repeated annealing treatments, such processing is set forth in G. Wiener patent application Serial No. 788,596, filed January 23, 1959, assigned to the assignee of the present invention. With this cyclic treatment of cleaning and reannealing, the cube grains can grow continually to almost the entire volume of the sheet. However, cube texture material made by secondary recrystallization techniques is invariably characterized by extremely large grain size which is generally not associated with optimum magnetic or mechanical properties.

The terms "primary recrystallization" and "secondary recrystallization" are used in the description of this invention in the same sense that they are understood and widely employed in the art. A very complete treatment of primary and secondary recrystallization in silicon iron alloys will be found in two published articles. The first article, entitled "Cold-Rolled and Primary Recrystallization Textures in Cold-Rolled Single Crystals of Silicon Iron," appears in Acta Metallurgica, vol. 2, March 1954, pages 174 to 183; and the second, entitled "On the Theory of Secondary Recrystallization Texture Formation in Face-Centered Cubic Metals," appears in Acta Metallurgica, vol. 2, May 1954, pages 386 to 393.

The cube texture can also be developed by cold rolling and annealing a grain oriented ingot or a slab cut therefrom, in which ingot columnar grains are produced to have a preferred orientation with a cube direction essentially parallel with the columnar axis. Such grain-oriented ingots are obtained by slow and careful unidirectional solidification of the ingot metal in a specially designed ingot mold, to which precisely controllable warming and cooling devices have been attached.

It is manifest that all the above processes require special and complicated devices, and critically controlled conditions. The principal effort of this invention is directed

to providing a commercially feasible process for making a cube texture magnetic material.

Accordingly, it is a general object of the present invention to provide a process for producing a ferrous-base intermediate material with a [001] fiber texture which is subjected to cold rolling and annealing to produce by primary recrystallization a soft magnetic polycrystalline sheet having essentially equal magnetic properties in the rolling direction and in the direction transverse thereto.

It is a more specific object of this invention to provide a method comprising at least two cold rolling and primary recrystallization anneal cycles to convert a sheet of iron-base alloy having a [001] fiber texture into a soft magnetic polycrystalline sheet having a high proportion of cube grains.

It is another object of this invention to provide an iron-base sheet material having a [001] fiber texture.

Another object of the invention is to provide a process for producing primary recrystallized magnetic sheet having a high proportion of cube-on-face grains, by cold rolling at least once a ferrous metal sheet having predominantly grains whose orientation is from (210) [001] to (100) [001] and subjecting the sheet to successive low temperature and high temperature primary recrystallization anneals.

Another object of the invention is to provide a process for producing a primary recrystallized sheet of ferrous magnetic material having over 50% of cube-on-face grains by cold rolling a sheet of material having predominantly grains whose orientation is from (410) [001] to (100) [001] and subjecting the sheet to successive low temperature and high temperature primary recrystallization anneals.

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 drawing, in which:

FIGURE 1 is a pole figure obtained from a [001] fiber texture sheet material; and,

FIG. 2 is a photomicrograph of a primary recrystallized cube texture material.

In accordance with the attainment of the foregoing objects, this invention is directed in one aspect to a method for making primary recrystallized cube texture iron-base alloy magnetic sheet from hot rolled plate having an essentially random grain orientation, but which may have a very weak preferred orientation due to the hot rolling to which it has been subjected. Derived from a hot rolled plate, an intermediate product of the method of this invention is a novel [001] fiber textured sheet material. More specifically, a hot rolled plate of iron-base alloy having essentially random grain or crystal orientation is subjected to the following process steps (1) annealing the plate at 800° C. to 1100° C. for a period of up to one hour, (2) pickling the plate in an acid solution to remove surface scale (these first steps have as their purpose to provide a clean plate of recrystallized ferrous metal. An anneal of one hour per inch of thickness is usually sufficient), (3) cold rolling the plate to effect a reduction in thickness of from 40% to 80% to form sheet material, (4) annealing the sheet in a wet hydrogen atmosphere at a temperature from 700° C. to 1000° C. for from one to five hours to provide for recrystallization and decarburization, and (5) annealing the recrystallized sheet at a temperature of from 1100° C. to 1300° C. for from 6 to 48 hours in dry hydrogen to effect secondary recrystallization, the above steps yielding a novel sheet material having a [001] fiber texture with the rolling direction as the fiber axis. The following additional process steps transform the [001] fiber texture sheet material into the cube texture final product: (6) cold rolling the fiber texture sheet material to a reduction in thickness of from 40% to 80%, (7) annealing the sheet material at a temperature in the range from 400° C. to 600° C. for from

about 4 to 48 hours to obtain partial primary recrystallization, (8) completing the annealing of the sheet material in the range from 700° C. to 1000° C. for a period of from ½ to 12 hours to obtain substantially complete primary recrystallization, (9) repeating the cold rolling and annealing steps (6), (7) and (8) at least once in the order set forth to produce primary recrystallized sheet material having at least 40% cube-on-face grains. For cases in which an increased grain size is desired, an additional final anneal may be applied to the sheet at a temperature in the range from 1100° to 1300° C.

After steps (6), (7) and (8) up to 25% or more of the grains have a (100) [001] texture with the majority of the remaining grains predominantly having orientations between (210) [001] and (100) [001]. Following the second cold rolling and primary recrystallization anneal of step (9) the sheet will comprise as much as 40% to 45% cube grains, with the majority of the remaining grains predominantly having orientations between (410) [001] and (100) [001]. After a third cold rolling and primary recrystallization anneal 70% to 75% or even more of the grains will exhibit (100) [001] orientation.

In the process of this invention, the [001] fiber texture sheet material is an intermediate product in the manufacture of cube texture sheet. However, it is not a transitory material, and it is therefore well suited for sale and use as an article of commerce. It may be employed for making magnetic cores. It also may be produced by one manufacturer for sale and shipment to a finishing manufacturer for processing into cube texture material.

It is noteworthy that prior to the process described herein, the term "fiber texture" has been appropriately applied only to material in wire form produced by drawing or extrusion. (See "Structure of Metals," first edition (1943), McGraw-Hill Book Company, Inc., by Charles S. Barrett, pages 381-382). Using the process of this invention a [001] fiber texture material is produced in sheet form for the first time within the knowledge of the inventor.

In general, [001] fiber texture sheet material may be transformed into commercially useful material having a predominant proportion of cube grains by from two to four cold rolling and primary recrystallization annealing cycles. For some applications an amount of cube texture as low as 40% has useful commercial value, and such a sheet material can be obtained from the [001] fiber texture sheet material with only two cold rolling and annealing cycles. The preferred cube texture sheet material, that is, one having from 70% to 75% cube grains, requires three such cycles. Even greater proportions of cube grains can be obtained by employing four such cycles.

The following materials are suitable for use in practicing this process: iron with up to 6% silicon, iron with up to 8% aluminum, and iron with up to 5% molybdenum. Iron may also be alloyed with combinations of two or more of silicon, aluminum and molybdenum. A small amount of carbon (preferably less than .005%) may also be present in these materials. More carbon can be tolerated, but this involves some sacrifice in magnetic properties. Small amounts of manganese, up to 1%, will usually be present in the alloy. Incidental impurities and small amounts of other elements may be present.

The [001] fiber texture is a texture in which the [001] direction is in the direction of rolling. In the [001] fiber texture there are large numbers of planes having orientations lying between the extremes (100) [001] orientation and (110) [001] orientation, the (110) [001] orientation being such that four cube faces are 45° away from the sheet surface and the desired (100) [001] orientation.

The following example illustrates the preparation of a [001] fiber texture sheet.

Example 1

The material used in this process was a 0.3 inch thick hot rolled plate of a commercial grade 3% silicon-iron

alloy having the following chemical composition in weight percent:

Si	C	Mn	P	S	Cu	Sn	O ₂	Sol. Al	Insol. Al	Iron
3.10	0.028	0.087	0.007	0.023	0.012	0.007	0.005	0.005	0.0029	Bal.

The plate was annealed at 1000° C. for twenty minutes in hydrogen followed by pickling in a sulfuric acid solution to remove surface scale. It was then cold-rolled to 0.100 inch. The cold-rolled strip was recrystallized and decarburized in a furnace at a temperature of 850° C. and was annealed at that temperature for 2 hours in a wet hydrogen atmosphere (80° F. dew point) primarily to decarburize the metal. The recrystallized strip was then annealed at 1200° C. for eight hours in dry hydrogen. The effect of the annealing treatment was a full secondary recrystallization. The grain size of the annealed strip was fairly uniform, and the average size was approximately 2 mm. in diameter.

Thirty grains were selected at random across the 2 inch width of the strip and the orientation of the grain was determined by an optical goniometer. As shown in FIG. 1, these grains had a range of orientations of the planes from (110) to (100) and essentially parallel with the plane of rolling, and a common [001] axis being nearly parallel with the rolling direction. Thus, the preferred grain orientation of the strip can be described as a [001] fiber texture with the rolling direction as the fiber axis.

In FIG. 1, the orientation (110) is indicated on the equator of the pole figure by the symbol $\circ$, the (210) orientation by the symbol $\times$, the (410) orientation by the symbol $\circ$, and the cube orientation by the symbol $\square$.

It should be understood that the process to produce the [001] fiber texture may be varied substantially, preferably the hot-rolled plate used as a starting material may have a thickness lying in the range from .050 inch to .60 inch. The initial anneal may be carried out at a temperature of from 800° C. to 1100° C. for periods of up to one hour or longer and other acids such as hydrochloric and nitric acid may be employed to pickle the surface oxides. In some cases, the hot rolled plate may have been annealed and pickled by the steel supplier. Therefore, it can be directly cold rolled without further treatment. The cold rolling of the annealed, cleaned plate should effect a reduction of from 40% to 80%; the annealing in wet hydrogen of a dew point of up to 150° F. may be carried out at a temperature of from 700° C. to 1000° C. for from about one to five hours; and the secondary recrystallization annealing in dry hydrogen may be carried out at a temperature of from 1100° C. to 1300° C. for a period of from 6 to 48 hours. Further, while the average grain size of 2 mm. in diameter as obtained in this example is good, an acceptable range for average grain size is from 0.5 to 5 mm.

The following examples illustrate the practice of the invention using the [001] fiber texture sheet as a starting material.

Example II

A 0.100 inch [001] fiber texture strip, obtained as described above, was cold rolled 40% to a thickness of 0.060 inch. It was then annealed at 550° C. for 24 hours to initiate primary recrystallization. The strip was then further annealed at 700° C. for two hours to complete primary recrystallization. The amount of cube grains after this anneal was approximately 17%. The strip was again annealed at 800° C. for two hours. The amount of cube grains increased from 17% to 25%. The remainder of the grain texture predominantly comprised grains having orientations ranging from (210) [001] to (100) [001]. The strip was then cold-rolled for the second time, with a reduction of 70% to a thick-

ness of 0.018 inch. After annealing for 24 hours at 550° C. and completing the primary recrystallization an-

nealing at 800° C. for two hours, the amount of cube grains had increased from 25% to 40%. The remainder of the grains had orientations predominantly ranging from (410) [001] to (100) [001]. The 0.018 inch thick strip was cold-rolled for the third time with a reduction of 70% to a thickness of approximately 0.006 inch. The specimen was then again annealed first for 24 hours at 550° C. and then at 800° C. for two hours to develop full primary recrystallization. All anneals were accomplished by charging the specimens into the furnace and heating the furnace and specimen together to the desired temperature. The final structure consisted of uniformly fine grains with an average diameter of 0.04 mm. The amount of cube grains was 70% and nearly all of the cube grains had two of their faces within 5° of the plane of the sheet. The distribution of cube edge direction among the cube grains, using the rolling direction as the reference, showed that 62% of the cube grains were within $\pm 5^\circ$, that 85% of the grains were within $\pm 10^\circ$, and that 90% of the grains were within $\pm 15^\circ$.

In the photomicrograph of FIG. 2, the light colored areas are the cube oriented grains. To obtain a product having optimum magnetic properties an additional anneal at a temperature higher than 800° C., preferably in the range from 1100° C.-1300° C., may be applied to the fully primary recrystallized sheet, to increase the grain size and improve the degree of crystal and orientational perfection of the cube grains. A 1200° C. anneal for 2 hours produced an increase in grain size. However, little or no secondary recrystallized grains will form during this last anneal.

All annealing in this process was carried out in a nickel-chromium alloy tube furnace with the dry hydrogen atmospheres having a dew point of about -60° C. The specimens were charged into the furnace at room temperature, and thereafter heated up to the indicated temperature. After the specimens were held at temperature for the time specified, they were removed to the cold zone inside the furnace tube for cooling.

Example III

A process essentially similar to that followed in Example II was employed; the process differing only in that the amount of reduction in the first cold-rolling step was increased. Thus, the same 0.100 inch thick [001] fiber texture strip was employed. The fiber texture strip was first cold-rolled 60% to a thickness of 0.040 inch. It was then annealed at 550° C. for 24 hours and primary recrystallization was completed by further annealing at 700° C. for two hours. The strip was further annealed at 800° C. for two hours. The strip was then cold-rolled for the second time, with a reduction of 70% to 0.012 inch thick. There followed an anneal for 24 hours at 550° C. and then at 800° C. for two hours. The strip was cold-rolled for the third time to effect a reduction of 70% to a thickness of approximately 0.004 inch. The final anneal was similar to that of Example II. The amount of cube grains was approximately from 70% to 75%, and the other structural features of the sheet were essentially the same in this process as in the process of Example II. As in Example II, an additional anneal of from 1100° C. to 1300° C. may be used to obtain a product having optimum magnetic properties.

While the Examples II and III show processes which have been successfully used to produce a cube texture

material from a [001] fiber texture material, it should be understood that the conditions and operations described in the examples may be varied somewhat within limits and still produce a superior cube texture material. For example, the amount of cold reduction may be varied within the range 40% to 80%.

An important feature of the invention is the primary recrystallization anneal which comprises the initial low temperature anneal to initiate recrystallization followed by anneal at 700° C. to 1000° C. to complete primary recrystallization. The temperature of the first anneal for initiating primary recrystallization may lie within the range of 400° C. to 600° C., and the time at the chosen temperature may vary from about 4 to 48 hours or even longer; however, the shorter times will be sufficient at the higher temperatures.

Since it is the low temperature anneal which initiates the desired primary recrystallization, some comment on the phenomena involved is warranted. In the study of recrystallization of single crystals, it has been observed that a (110) [001] crystal after cold rolling and primary recrystallization anneal, can form either in the (110) [001] orientation or in the (210) [001] orientation depending upon the temperature of the anneal. Thus, if the rolled crystal is annealed at a relatively high temperature a (110) [001] orientation will be obtained. On the other hand, if a relatively low temperature is initially employed, the orientation obtained will be (210) [001].

Similarly, a (210) [001] crystal can form, after cold rolling and primary recrystallization anneal, either in the (210) [001] orientation, if a high temperature anneal is employed, or in the (410) [001] orientation, if a low temperature anneal is employed.

When cold rolling and a primary recrystallization anneal are applied to a crystal having a (410) [001] orientation, the primary recrystallization product ordinarily will have a (100) [001] orientation because the (410) [001] orientation is already quite close to the (100) [001] orientation. The (100) [001] orientation is, of course, the cube-on-face orientation.

From the above experimental observations, it is clear that a crystal having an original orientation of (110) [001] can be transformed to a product having a cube orientation by successive cold rolling and primary recrystallization anneal cycles, provided that a low anneal temperature is used at least during the initial portions. If a high temperature anneal alone is employed, the product after the cold rolling and primary recrystallization anneal will have essentially the same orientation as the original crystal.

In the process of this invention, the above principles are applied to polycrystalline material having a [001] fiber texture. The [001] fiber texture material, as indicated previously, comprises a range of orientations from (110) [001] to (100) [001] including the orientations (210) [001], (410) [001], and innumerable intermediate orientations. The [001] fiber texture material is cold rolled producing numerous deformation texture components having different recrystallization temperatures. The low temperature primary recrystallization anneal promotes the recrystallization of certain deformation texture components which give rise to grains having desired preferred orientations. At the same time, this low temperature anneal tends to suppress the recrystallization of other deformation texture components which, if recrystallized, would give rise to grains of undesired orientations. The crystals in the polycrystalline material having the orientations (110) [001], (210) [001], and (410) [001] apparently respond to the cold rolling and primary recrystallization anneal in the manner predicted from the experiments on single crystals, that is, by rotating along an axis parallel to the direction of rolling successively closer to cube-on-face orientation on each cycle of cold rolling and annealing. Further, it appears that the crystals having orientations intermediate the principal orientations named, upon

recrystallization, also tend to approach the cube orientation. Once a grain has reached a cube-on-face orientation, it retains this orientation through further cold rolling and annealing steps.

Once the growth of the desired grain nuclei is well initiated, by the low temperature anneal, the temperature is raised to promote rapid growth of the grains having the desired orientations. This latter temperature may lie in the range from 700° C. to 1000° C. for from ½ to 12 hours, and is maintained until full primary recrystallization is attained. This latter anneal temperature may be varied. Thus, the sheet may be annealed 2 hours at 800° C. followed by 2 hours at 900° C.

A particularly notable feature of the process of this invention, when contrasted with the processes of the prior art for obtaining preferred orientations in magnetic materials, lies in the non-criticality of the furnace atmosphere. As a matter of convenience, dry hydrogen was used as the atmosphere in the examples given, however, wet hydrogen, ammonia, inert gases such as argon, and even air may be used without adversely affecting the texture of the material. Of course, if the atmosphere used permits as undesirable degree of oxidation to occur, the products of oxidation must be removed before the sheet material may be used commercially. To avoid this additional processing step then, and because hydrogen gas is usually readily available in plants producing materials of this kind, as a practical matter, an atmosphere of hydrogen will be used.

While the product made in the examples was of a thin gauge, that is 0.004 inch to about 0.006 inch, the strip may be made in thicknesses substantially greater. The principal limitations on thickness is the result of the practical difficulty involved in cold rolling extremely thick plates.

The processes of the examples employed an iron silicon alloy. However, the process will work equally well on iron-aluminum, and iron-molybdenum alloys. All of these materials have essentially similar rolling textures and similar annealing textures. Iron with up to 6% silicon, iron with up to 8% aluminum, and iron with up to 5% molybdenum, may all be used in the practice of this invention. Iron with two or more of silicon, aluminum, and molybdenum may also be used. For example, an alloy comprising 3% silicon, 0.5% molybdenum and the balance iron may be employed.

As the process has been described a hot-rolled plate having an essentially randomly oriented crystallographic structure is treated to produce a cube texture material. As an essential intermediate product a [001] fiber texture material is first produced. Of course, if a fiber texture sheet material is available, only the novel cold-rolling and annealing steps need be applied thereto.

As indicated previously, the first cold rolling and primary recrystallization anneal on the [001] fiber texture material produces a material which contains from 17% to 25% cube grains with the balance of the grains having orientations lying predominantly in the range from (210) [001] to (100) [001] (See FIGURE 1). When a material having this latter range of orientations, no matter how produced, is subjected to the cold rolling-annealing cycle of this invention, a sheet material will be produced having 40% or more cube grains with the balance of the grains having orientations lying predominantly in the range from (410) [001] to (100) [001]. And when this latter material is subjected to the cold rolling-annealing cycle a material having grains predominantly of cube orientation is produced.

Materials having essentially (210) [001] to (100) [001] orientations produced by casting techniques or by single crystal techniques or other rolling and annealing practices need be subjected only to two cold rolling and annealing cycles to produce material having a predominantly cube texture. Similarly, material produced by these last techniques having (410) [001] to (100) [001] orientations need be subjected only to one cold rolling

and annealing cycle to produce material having essentially cube texture.

There has thus been presented a relatively simple process for producing a cube texture magnetic material which operates by the controlled selected primary recrystallization and growth among the various texture components of fiber textured material. Further, this technique relies upon primary recrystallization rather than secondary recrystallization as is common in the art. The primary recrystallization technique is advantageous in that it permits relatively easy control of grain size, it eliminates critical furnace atmospheres, lowers the annealing temperatures required, and provides stronger sheet material. Further, since the processing conditions are less critical than in prior art processes, stress relief anneal after punching may be carried out without the highly critical attention to processing conditions commonly required in the prior art.

The inventive principles embodied in the above description may obviously be incorporated in modified processes by those skilled in the art without departing from the spirit and scope of this invention, and it is intended that the description be interpreted as illustrative and not in a limiting sense.

I claim as my invention:

1. A process for producing cube texture iron-base sheet material for magnetic purposes from hot-rolled plate having a recrystallized structure and essentially random grain orientation, comprising the steps of, (1) cold rolling the plate from 40% to 80% to form sheet material, (2) annealing the reduced sheet in a wet hydrogen atmosphere at a temperature of from 700° C. to 1000° C. for from about 1 to 5 hours to provide recrystallization and decarburization, (3) annealing the recrystallized sheet at a temperature of from 1100° C. to 1300° C. for from 6 to 48 hours in dry hydrogen, the above steps yielding a sheet material having a [001] fiber texture with the rolling direction as the fiber axis, (4) cold rolling the fiber texture sheet to effect a reduction of from 40% to 80%, (5) annealing the material at a temperature in the range from 400° C. to 600° C. for from about 4 to 48 hours to obtain partial primary recrystallization, (6) completing the annealing of the sheet material at temperatures in the range from 700° C. to 1000° C. for a period of from about ½ to 12 hours to obtain substantially complete primary recrystallization, and (7) repeating steps 4, 5 and 6, at least once in the order set forth to produce a fine-grained sheet material having at least 40% cube grains, the iron-base sheet material comprising at least one element selected from the group consisting of silicon, aluminum, and molybdenum, the silicon not exceeding 6%, the aluminum not exceeding 8%, and the molybdenum not exceeding 5%, and the balance being iron with small amounts of incidental impurities.

2. The process of claim 1 in which increased grain size is produced by utilizing an additional final anneal at a temperature of from about 1100° C. to 1300° C.

3. A process for producing cube texture iron-base sheet material for magnetic purposes from hot-rolled plate having a recrystallized structure and essentially random grain orientation, comprising the steps of, (1) cold rolling the plate from 40% to 80% to form sheet material, (2) annealing the reduced sheet in a wet hydrogen atmosphere at a temperature of from 700° C. to 1000° C. for about 1 to 5 hours to provide recrystallization and decarburization, (3) annealing the recrystallized sheet at a temperature of from 1100° C. to 1300° C. for from 6 to 48 hours in dry hydrogen, the above steps yielding a sheet material having a [001] fiber texture with the rolling direction as the fiber axis, (4) cold rolling the fiber texture sheet to effect a reduction of from 40% to 80%, (5) annealing the material at a temperature in the range from 400° C. to 600° C. for from about 4 to 48 hours to obtain partial primary recrystallization, (6) completing the annealing of the sheet material at temperatures in the range from 700° C. to 1000° C. for a period of from

about ½ to 12 hours to obtain substantially complete primary recrystallization, and (7) repeating steps 4, 5 and 6, at least twice in the order set forth to produce a relatively fine-grained sheet material having at least 50% cube grains, the iron-base sheet material comprising at least one element selected from the group consisting of silicon, aluminum, and molybdenum, the silicon not exceeding 6%, the aluminum not exceeding 8%, and the molybdenum not exceeding 5%, and the balance being iron with small amounts of incidental impurities.

4. A process for producing [001] fiber texture iron-base sheet material from hot rolled annealed and cleaned plate having essentially random grain orientation, comprising the steps of, (1) cold rolling the hot rolled plate from 40% to 80% to form sheet material, (2) annealing the reduced sheet in a wet hydrogen atmosphere at a temperature of from 700° C. to 1000° C. for from 1 to 5 hours to provide recrystallization and decarburization, (3) annealing the recrystallized sheet at a temperature of from 1100° C. to 1300° C. for from 6 to 48 hours in dry hydrogen, thereby producing a sheet material having a [001] fiber texture with the rolling direction as the fiber axis, the iron-base sheet material comprising at least one element selected from the group consisting of silicon, aluminum and molybdenum, the silicon not exceeding 6%, the aluminum not exceeding 8%, and the molybdenum not exceeding 5%, and the balance being iron with small amounts of incidental impurities.

5. A process for producing highly grained oriented iron-base sheet material for magnetic purposes from a rolled material having a [001] fiber texture with the rolling direction as the fiber axis, comprising the steps of, (1) cold-rolling the fiber texture sheet material to a reduction of from 40% to 80%, (2) annealing the sheet material at a temperature in the range from 400° C. to 600° C. for from about 4 to 48 hours to obtain partial primary recrystallization, (3) completing the annealing of the material at a temperature in the range of from 700° C. to 1000° C. for a period of ½ to 12 hours to obtain substantially complete primary recrystallization, the material then having a high proportion of grains with orientations ranging from (210) [001] to (100) [001], (4) repeating the cold rolling and annealing steps (1), (2) and (3) to produce material having a high proportion of grains with orientations ranging from (410) [001] to (100) [001], (5) repeating the cold rolling and annealing steps (1), (2) and (3) to produce material having a fine grain size and 50% and more of the grains with (100) [001] orientation, the iron-base sheet material comprising at least one element selected from the group consisting of silicon, aluminum, and molybdenum, the silicon not exceeding 6%, the aluminum not exceeding 8%, and the molybdenum not exceeding 5%, and the balance being iron with small amounts of incidental impurities.

6. The process of claim 5 in which increased grain size is produced by utilizing an additional final anneal at a temperature of from 1100° C. to 1300° C.

7. A process for producing cube textured material from iron-base sheet material having a major proportion of grain orientations ranging from (210) [001] to (100) [001], the steps comprising, (1) cold rolling the sheet material to a reduction of from 40% to 80%, (2) annealing the sheet at a temperature in the range from 400° C. to 600° C. for from 4 to 48 hours to obtain partial primary recrystallization, (3) annealing the material at a temperature in the range from 700° C. to 1000° C. for a period of from ½ to 12 hours to obtain substantially complete primary recrystallization, the sheet then comprising a material having approximately 40% cube texture and the balance a range of orientations from (410) [001] to (100) [001], (4) repeating the cold rolling and annealing steps (1), (2) and (3) to produce material having a fine grain size and 70% or more of the grains with (100) [001] orientation, the iron-base sheet material comprising at least one element selected from the

group consisting of silicon, aluminum, and molybdenum, the silicon not exceeding 6%, the aluminum not exceeding 8%, and the molybdenum not exceeding 5%, and the balance being iron with small amounts of incidental impurities.

8. The process of claim 7 in which increased grain size is produced by utilizing an additional final anneal at a temperature of from about 1100° C. to 1300° C.

9. A process for producing a fine-grained cube textured material from iron-base sheet material having a major proportion of grains with orientations ranging from (410) [001] to (100) [001], the steps comprising, (1) cold rolling the sheet material to a reduction of from 40% to 80%, (2) annealing the sheet at a temperature in the range from 400° C. to 600° C. for from 4 to 48 hours to obtain partial primary recrystallization, (3) completing the annealing of the material at a temperature in the range of from 700° C. to 1000° C. for a period of from about ½ to 12 hours to obtain substantially complete primary recrystallization, the sheet then consisting of a material having over 50% of the grains with (100) [001] orientation, the iron-base sheet material comprising at least one element selected from the group consisting of silicon, aluminum, and molybdenum, the silicon not exceeding 6%, the aluminum not exceeding 8%, and the molybdenum not exceeding 5%, and the balance being iron with small amounts of incidental impurities.

10. The process of claim 9 in which increased grain size is produced by utilizing an additional anneal at a temperature of from 1100° C. to 1300° C.

11. In a process for producing cube texture iron-silicon sheet material suitable for magnetic applications comprising, by weight, silicon in an amount not exceeding 6%, and the balance essentially iron with small amounts of incidental impurities, from a hot rolled plate of the material having a recrystallized structure and essentially

random grain orientation, the steps of, (1) cold rolling the recrystallized plate to effect a reduction of from 40% to 80% to form a sheet, (2) annealing the reduced sheet in a wet hydrogen atmosphere at a temperature of from 700° C. to 1000° C. for from about 5 to 1 hours to provide recrystallization and decarburization, (3) further annealing the recrystallized sheet at a temperature of from 1100° C. to 1300° C. for from 6 to 48 hours in dry hydrogen, the above steps yielding a sheet having a [001] fiber texture with the rolling direction as the fiber axis, (4) cold rolling the fiber texture sheet to effect a reduction of from 40% to 80%, (5) annealing the sheet at a temperature in the range from 400° C. to 600° C. for from about 4 to 48 hours to initiate primary recrystallization, (6) completing the annealing of the sheet material at temperatures in the range from 700° C. to 1000° C. for a period of from about ½ to 12 hours to obtain substantially complete primary recrystallization, and (7) repeating steps (4), (5) and (6) at least once in the order set forth to produce a primary recrystallized, fine grain sheet material having at least 40% by volume of cube-on-face grains.

12. The process of claim 11 in which an increased primary recrystallized grain size is produced by utilizing an additional anneal at a temperature of from 1100° C. to 1300° C.

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