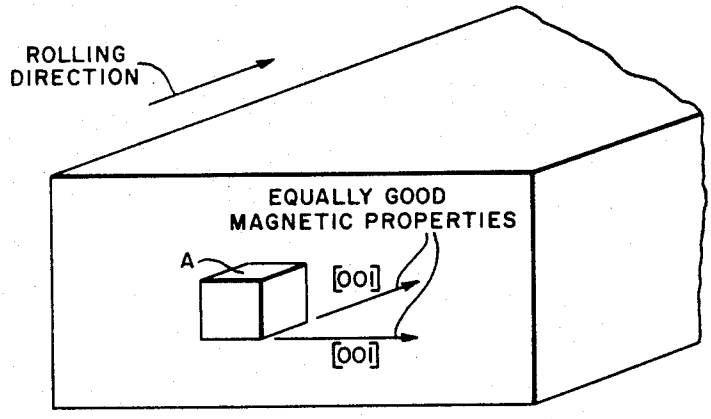
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HEAVY GAUGE DOUBLE ORIENTED MAGNETIC SHEET MATERIAL

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A-CUBE ON FACE OR DOUBLE ORIENTATION

WITNESSES

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HEAVY GAUGE DOUBLE ORIENTED MAGNETIC SHEET MATERIAL

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This invention relates to the field of soft magnetic materials and is particularly directed to a process for making polycrystalline molybdenum containing iron-base alloy sheet material having a highly developed cube-texture.

The soft magnetic materials, such as iron and its alloys, have found wide industrial application as core materials in power transformers, motors, generators, and in other applications. These cores are usually laminated structures made by stacking relatively thin sheets of the 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 and iron-base alloys, much work has been done to obtain preferred orientation of the crystals in polycrystalline material. Iron and iron-base alloys have a body-centered cubic crystalline structure at low temperatures and retain this 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.

It is highly desirable to have available a sheet of material having a large proportion of the grains thereof arranged to provide optimum magnetic properties in two directions, known as cube-on-face or cubic texture and which may be described in the Miller Indices 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 or the edges of the sheet, 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.

It has long been recognized for the above and other reasons that a polycrystalline sheet material of iron or

iron-base alloys having the cube-on-face grain orientation such that the optimum magnetic properties, substantially equal in a longitudinal and transverse direction, and comparable to those previously available only in the rolling direction for cube-on-edge grain orientation 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 were available.

Most of the work which has been done in the preparation of cube texture magnetic materials has been directed to the silicon-iron family of alloys. In thin gauge, 5 mils and less, silicon-iron sheets, it has been readily possible to obtain a high proportion of area of cube-on-face grains. In copending application Serial No. 722,778, filed March 20, 1958, now abandoned, and assigned to the assignee of the present invention, there is described a process for obtaining cube texture in relatively thick silicon-iron alloy sheets. While a high degree of double orientation is achieved by the process described in that application and, hence, a very useful material is produced, the proportion of area comprising doubly oriented grains is not as great as in the extremely thin silicon-iron alloy sheets.

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. II, March 1954, pages 174–183; and the second entitled "On the Theory of Secondary Recrystallization Texture Formation in Face-Centered Cubic Metals" appears in Acta Metallurgica, vol. II, March 1954, pages 386–393.

The object of the invention is to provide a process for cold working and heat treating magnetic molybdenum-iron alloys so as to produce relatively heavy gauge cube texture sheet.

Another object of the invention is to provide iron-molybdenum alloys that respond to cold working, an intermediate heat treatment, and a final heat treatment to produce growth of doubly oriented cube-on-face grains in magnetic sheets, which treatment is particularly effective in gauges from 8 to 25 mils.

It is another object of the invention to provide iron-molybdenum alloys containing a small amount of silicon therein, that respond to cold working, an intermediate heat treatment, and a final heat treatment to produce growth of double oriented cube-on-face grains in magnetic sheets particularly in gauges of from 8 to 25 mils.

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 drawings, in which:

The single figure is a schematic view in perspective illustrating the desired grain orientation in the magnetic sheets.

Referring to the drawing, there is illustrated a sheet of metal in which is schematically depicted a cube A which comprises a cube-on-face or double oriented grain. The cube A, it will be noted, stands with two faces parallel to the plane of the rolled surface of the sheet. Four edges of the cube A are aligned parallel to the rolling direction. The direction of easiest magnetization of the cube is along the cube edges or the [001] direction. The cube A has four cube edges oriented in the direction of rolling and four cube edges oriented in the transverse direction. The optimum magnetic properties are obtained in both the rolling direction and in the transverse direc-

tion. Magnetic sheets comprising predominantly grains oriented in the manner of cube A will similarly have optimum magnetic properties in the rolling direction and in the direction transverse thereto.

It has been discovered that cube-on-face or double oriented 3% to 5% molybdenum-iron magnetic sheets having a major proportion of its area of (100) [001] grain texture may be readily produced to a gauge of from 8 to 25 mils from hot rolled molybdenum-iron alloy sheet material. Thinner gauge sheet may be also produced with cube texture. The process requires at least two cold rolling steps each of which affects a deformation of at least 30% and not more than 80% in thickness with an intermediate critical anneal of the cold reduced material to effect complete primary recrystallization followed by a final anneal which results in first producing oriented nuclei in the sheet and then causing the nuclei to grow through the heavy gauge material by a secondary recrystallization process.

More specifically, the alloy material in flat or ingot form is hot rolled at a temperature in the range from 900° C. to 1200° C. to a thickness of from 0.15 to 0.5 inch, the hot rolled sheet is then cold rolled with a reduction of from 30% to 80%, and then the cold rolled sheet is annealed at a temperature in the range from 700° C. to 1100° C. in an atmosphere of dry hydrogen having a dew point of at least —30° C., the annealed sheet is subjected to a second cold rolling step to produce a reduction of from 30% to 80%, the rolled sheet is again annealed for a period of about 2 hours at a temperature of from 900° C. to 1200° C. in an atmosphere of dry hydrogen having a dew point of at least —40° C., and then a final cold rolling step effects a reduction of from 30% to 80%. This rolled sheet is then finally annealed to effect substantially complete recrystallization at a temperature of from 1050° C. to 1400° C. in an atmosphere of dry hydrogen having a dew point of at least —40° C., or a vacuum of at least 10^{-1} mm. of mercury. The period for the final anneal may be from 2 to 16 hours at 1200° C., lesser times being acquired at higher temperatures. It has thus been discovered that heavy gauge molybdenum-iron magnetic sheet, that is, from about 8 mils to about 25 mils, will result, in which more than 90% of the area of the sheet comprises grains so oriented that they have a (100) plane lying within 10° of the plane of the sheet, while more than 80% of the area comprises cube-on-face grains are so oriented that they have a [100] direction or edge within 10° of the rolling direction or edge of the sheet.

The process of the present invention is applied to sheets of molybdenum-iron alloys containing from 3% to 5% by weight of molybdenum and the balance being iron except for small amounts of incidental impurities, or to molybdenum-silicon-iron alloys containing from 3% to 5% by weight of molybdenum, from 0.5% to 1.5% silicon, with the balance iron except for small amounts of incidental impurities.

The process is illustrated by the following examples. Small amounts of additives such as manganese, up to 2%, and aluminum, up to 0.05%, may be present.

The following ferrous base alloys were vacuum melted in magnesia crucibles using grade AlO_4 electrolytic iron:

(1) 4% molybdenum-iron
(2) 4.5% molybdenum-iron
(3) 3% molybdenum—1% silicon-iron
(4) 4% molybdenum—0.5% silicon-iron

After melting was completed, each alloy was poured into a stainless steel slab mold to produce an ingot of approximately 5 pounds, which is the typical size of ingots employed for carrying on such investigations.

The following processes were employed in the treatment of certain of the alloys:

PROCESS A

In this process the alloy ingot is hot rolled at a temperature of about 1000° C. to a slab about 0.100 inch in thickness. The slab is then preferably treated to remove surface oxides by any of the well known procedures. The removal of oxides immediately after the hot reduction is not vital; however, it is desirable to remove them before the final cold reduction. The most commonly practiced process for removing the oxides is pickling in an acid. Sulfuric and hydrochloric acids were found to be suitable for the pickling.

After the slab has been pickled to remove the oxides, it is cold rolled to a sheet about 0.070 inch in thickness. The sheet is then annealed for two hours at about 1000° C. in an atmosphere of dry hydrogen having a dew point of at least —30° C. The sheet is then cold rolled from 0.070 inch to 0.030 inch. A last intermediate anneal is then given the sheet by holding it for two hours at 1000° C. in an atmosphere of dry hydrogen having a dew point of at least —50° C. The last intermediate anneal is followed by the final cold rolling step which reduces the sheet from 0.030 inch to 0.012 inch. A final anneal for 16 hours at 1200° C. in dry hydrogen of a —50° C. dew point was given the sheets and effected substantially complete secondary recrystallization.

PROCESS B

In this process the ingot is hot rolled at about 1000° C. to a slab of a thickness of about 0.250 inch. The slab is then pickled as described in process A to remove oxides. The oxide-free slab is cold rolled to reduce it from a thickness of about 0.250 inch to 0.100 inch. This is a 60% reduction in thickness. After the 60% cold-rolling deformation, the slab is annealed for about two hours at a temperature of about 800° C. in dry hydrogen at a dew point of —30° C. After the annealing step the slab is again cold rolled to reduce it from a thickness of about 0.100 inch to 0.040 inch. After the second deformation, the slab is again annealed for about two hours at about 1100° C. in dry hydrogen at a dew point of —50° C. Following the second anneal, the slab is cold rolled, reducing it from about 0.040 inch to 0.012 inch. By this three-step cold-rolling process a sheet 12 mils in thickness is thus produced. The sheets made by the above processing schedule were given a final anneal for 16 hours at 1200° C. in dry hydrogen having a dew point of at least —50° C. to effect substantially complete secondary recrystallization.

In the above processing methods, the intermediate anneals in dry hydrogen keep the surfaces of the sheet clean. The heat treatments were carried out in Inconel boats in an Inconel tube furnace. The sheets were separated by alumina powder.

Heat treated sheets made in accordance with the above processes were macroetched to determine the extent to which cube growth took place. Substantially all the sheet comprised secondary grains. Other samples were electropolished and domain patterns were observed on them to determine directional orientation. The results obtained for alloy 1 are given in Table I below:

Table I

ORIENTATION OF GRAIN IN 4% MOLYBDENUM IRON (ALLOY 1)

Process	Final Sheet Thickness, inches	Grains with (100) Plane Within 5° of Sheet Surface, percent	Directional Orientation: Grains With <100> Within Given Angle from Rolling Direction, percent			
			5°	10°	15°	20°
A	.012	95	41	70	87	95
B	.012	95	70	89	100	100

From the above table it is seen that both processes produce a sheet material with a very high degree of

cube growth, but differ significantly in the perfection of the directional orientation obtained, with process B showing a clear superiority in the alignment of cube edges with the rolling direction.

By way of comparison between the molybdenum iron alloys of this invention and a good silicon iron alloy, both alloys having a high degree of secondary recrystallization cube grain growth, Table II below sets forth pertinent data.

Table II

PROPERTIES OF DOUBLY ORIENTED ALLOYS

[0.012" thick sheets, more than 90% of secondary grains in {100} plane]

	Directional Orientation				D.-C. Magnetic Properties		
	Grains with <100> Direction Within Given Angle from Rolling Direction, percent				Coercive Force (Oersteds)	Induction at 10 Oersteds (Gausses)	Maximum Permeability
	5°	10°	15°	20°			
3% Si-Fe [1]	40	62	84	90	.072	16,800	35,500
4% Mo-Fe [2]	70	89	100	100	.055	18,600	55,300

[1] Process A (An optimum process for silicon-iron alloys).
[2] Process B.

Table II clearly illustrates the higher degree of directional orientation obtained with the heavy gauge magnetic sheet of this invention when compared with heavy gauge silicon-iron alloy sheet having essentially the same amount of cube growth.

Alloy 2 with 4.5% molybdenum, exhibited essentially the same characteristics as alloy 1 with respect to both cube growth and direction orientation when made in accordance with process B. Alloys 3 and 4 did not exhibit as complete cube growth in 0.012 inch thick sheet as alloys 1 and 2, but had very nearly as high a percentage of cube grain directional orientation as alloy 1. Alloy 3 exhibited a 60% of its area of cube grain growth while alloy 4 exhibited an 80% of its area of cube growth.

Thinner gauge sheets i.e. below 8 mils thickness, for example, 0.5, 1 and 2 mils thickness, with equally good cubic texture can be produced from the alloys of this invention by following the process here disclosed.

There has thus been set forth novel molybdenum iron alloys and a process for producing relatively thick magnetic sheets which are particularly useful for motor and generator cores Since certain changes in carrying out the above process may be made without departing from the scope of the invention, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

I claim as my invention:

1. A double oriented magnetic sheet comprising a molybdenum-iron alloy containing from 3% to 5% molybdenum and the remainder iron except for incidental impurities, the sheet having a thickness of up to 8 mils to 25 mils, the sheet comprising at least 90% by volume of secondary recrystallized grains having an orientation such that a (100) plane is within 10° of the plane of the sheet and at least 85% of these planes having a [001] direction within 10° of the direction of rolling.

2. A double oriented magnetic sheet comprising a molybdenum-silicon-iron alloy containing from 3% to 5% molybdenum, from 0.5% to 1.5% silicon, and the remainder iron except for incidental impurities, the sheet having a thickness of from 8 mils to 25 mils, the sheet comprising at least 60% by volume of secondary recrystallized grains having an orientation such that a (100) plane is within 10° of the plane of the sheet, and at least 85% of these planes having a [001] direction within 10° of the direction of rolling.

3. A double oriented mgnetic sheet comprising a molybdenum-iron alloy containing about 4% molybdenum and the remainder iron except for incidental impurities, the sheet having a thickness of from 8 mils to 25 mils, the sheet comprising at least 90% by volume of secondary recrystallized grains having an orientation such that a (100) plane is within 10° of the plane of the sheet and at least 85% of these planes having a [001] direction within 10° of the direction of rolling.

References Cited in the file of this patent

UNITED STATES PATENTS

2,867,558	May	Jan. 6, 1959
2,867,559	May	Jan. 6, 1959
2,940,881	Hollomon	June 14, 1960
2,992,951	Aspden	July 18, 1961
2,992,952	Assmus et al.	July 18, 1961