Jan. 18, 1955 C. G. DUNN 2,700,006

PROCESS FOR PRODUCING FINE-GRAINED HIGHLY ORIENTED SILICON STEEL

Filed Sept. 3, 1953 2 Sheets-Sheet 1

Fig. 1.

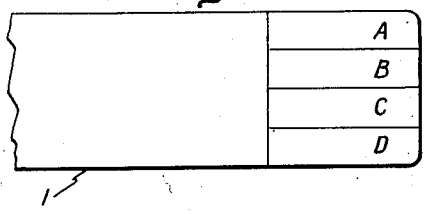


Fig. 2.

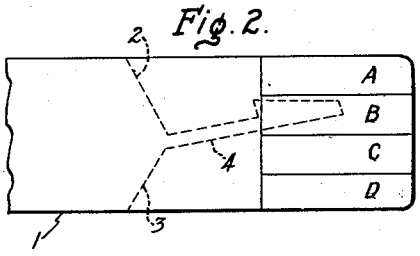


Fig. 3.

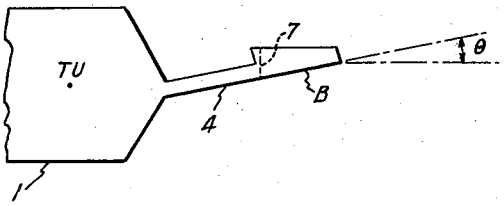


Fig. 4.

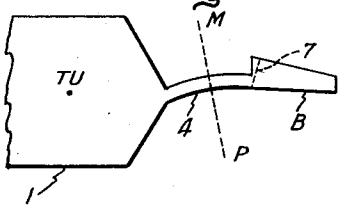


Fig. 5.

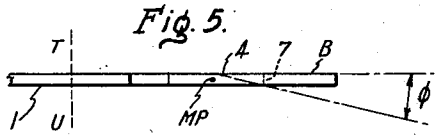


Fig. 6.

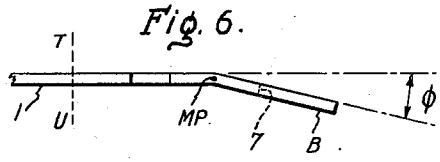


Fig. 7.

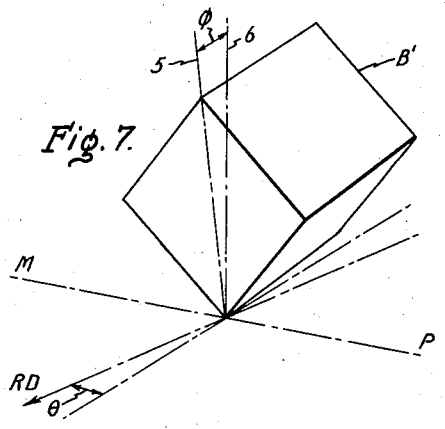


Fig. 8.

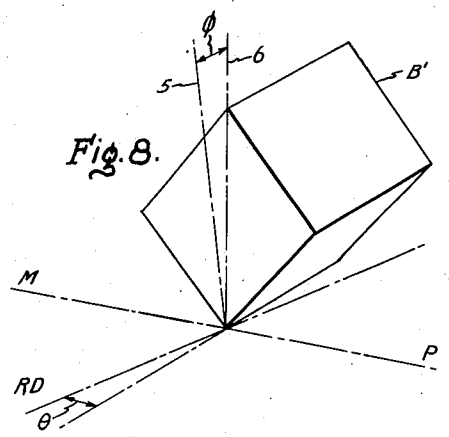
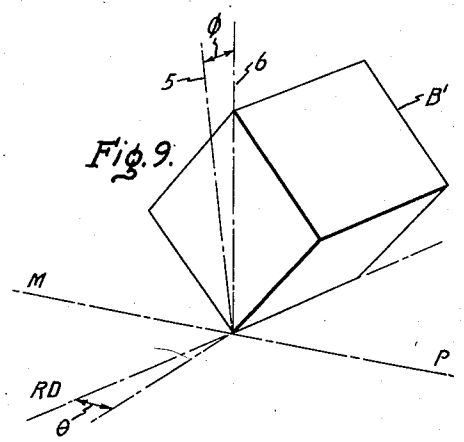


Fig. 9.



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Fig. 10

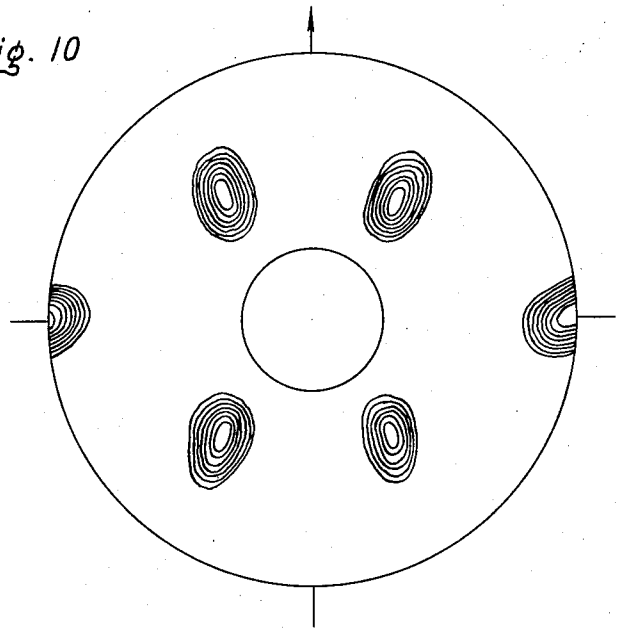
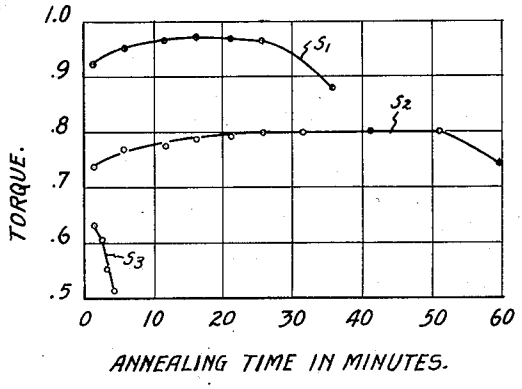


Fig. 11.



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PROCESS FOR PRODUCING FINE-GRAINED HIGHLY ORIENTED SILICON STEEL

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Application September 3, 1953, Serial No. 378,311

5 Claims. (Cl. 148—12.5)

This invention relates to magnetic materials. More particularly, it relates to a method of producing silicon steel magnetic strip material having a very high degree of preferred orientation.

Because of the anisotropic character of magnetic permeability u with crystallographic direction in single crystals of iron and other magnetic materials, it is desirable to align certain crystallographic directions of the crystals in any such material in such manner as to give the highest possible permeability u for a given flux density B or conversely to attain as high a flux density B as possible for a given applied magnetic field H where $B=uH$.

It is an object of this invention to provide a process for making magnetic strip material having a very high degree of preferred orientation.

Another object of the invention is to provide a process wherein the magnetic orientation of an entire strip of silicon steel is modeled after a single seed crystal.

A further object is to provide very thin strips of highly oriented magnetic material.

Other objects will become apparent from a consideration of the following description and the drawings in which Fig. 1 represents a number of crystals grown in one end of a silicon steel strip; Fig. 2 illustrates a method of isolating a seed crystal; Figs. 3–6 illustrate the reorientation of an actual seed crystal; Figs. 7 to 9 depict the reorientation of an idealized cubic crystal; Fig. 10 is a quantitative pole figure of a specimen having a desired orientation after a one minute anneal of 980° C.; and Fig. 11 is a graph giving the results of a magnetic torque test of three differently oriented specimens subjected to the same annealing treatment.

It is known that large crystals may be produced by feeding a metal, which has preferably been strained in tension, slowly into a furnace whose temperature is high enough to produce recrystallization in the sample. Likewise, different methods of rolling and heat treating magnetic steels have resulted in varying degrees of success in aligning the metallic grains of metal in such directions crystallographically as to give better magnetic properties. An example of the latter is the orientation (110) [100] for silicon steel cold rolled strip, the (110) notation denoting a (110) crystallographic plane in the plane of the piece and the [100] notation denoting a crystallographic direction of the form [100] parallel to the rolling direction. The ultimate favorable magnetic condition would be attained in material comprising a single crystal which is oriented in a selected and predetermined manner. In commercial production this single crystal is difficult to obtain due to the fact that upon recrystallization crystals having a variety of orientations are formed.

While fairly thick strips of single crystal material have been formed, attempts to produce long strips of single crystal material thinner than 0.005 inch in thickness have been unsuccessful.

It has been found that if a single crystal of silicon steel strip is cold rolled and recrystallized under carefully controlled conditions the final magnetic orientation of the strip is no longer that of a single crystal but approaches very closely to what it would be if the entire strip were composed of a single properly oriented crystal.

Briefly stated, in accordance with one of its aspects, this invention comprises isolating a crystal at one end of a single silicon steel strip, cutting away other crystals from this end to leave the crystal connected by a thin neck to the remainder of the strip, orienting the crystallographic directions of the crystal into (110) [001] orientation by plastically deforming the neck at red heat, introducing the end of the strip including the seed crystal into a temperature gradient furnace to heat the strip to a temperature of 900°–1000° C., moving the strip into the furnace at a rate equal to the rate of growth of the crystal, cold rolling the strip to bring about a reduction in thickness of about 70 per cent, and recrystallizing the strip at a temperature of about 980° C. in a reducing atmosphere.

It has been found that the conditions for transforming a piece of magnetic material into a single crystal of desirable orientation and subsequently converting a large portion of strip material to which the crystal is attached to approximately the orientation of the seed crystals are as follows: First, the matrix or material to be transformed must be able to support the growth of a grain or crystal of the specified orientation. Second, one or more grains must be grown at one end of the strip and a seed crystal selected and isolated from the rest of the strip except for a small neck or matrix material which connects it to the main part of the strip. Further, the seed crystal must be placed in the desired orientation with respect to the strip proper. Third, the sheet or strip with the reoriented seed crystal must be fed into a furnace at a speed corresponding to the natural rate of growth of the selected seed crystal for a temperature as high as possible in a safe range where other grains will not have time to start their growth. Fourth, the single crystal thus produced is cold rolled to reduce its thickness by about 70 per cent. Fifth, the strip is subjected to a recrystallization treatment at a temperature of about 980° C.

In carrying out the invention, strip material of any convenient thickness may be used as a starting material. The end of the strip **1** in the drawing is placed in a furnace to grow a number of grains such as A, B, C, and D in one end thereof. Upon the basis of X-ray analysis a crystal such as B is selected as the seed crystal. The sample is then cut as indicated by dotted lines **2** and **3** in Fig. 2 leaving the open grain boundary **7** of B in the sample connected to the main sample by a neck **4**. This cutting may be accomplished by etching with a suitable reagent using a stopoff lacquer to control the process, or, when done mechanically, residual edge strains in the sample left by cutting may be removed by etching off about one mil of the edges by an etching treatment, the faces of the sample being protected by a layer of lacquer.

Having isolated a good seed crystal B as shown in Fig. 3, the next step is to place the crystal B in any desired orientation with respect to the main strip by deforming or twisting neck **4** at red heat according to the change in orientation required. In carrying out the deformation, it is preferred to reorient crystal B by making two successive angular rotations of neck **4** at red heat, one rotation being about an axis TU normal or perpendicular to the plane of the strip or sample **1** and the other rotation being about an axis MP lying in the plane of the strip. The axis MP is chosen in advance of reorientation according to the original orientation of the crystal and the final orientation desired and need not be perpendicular to the long direction or rolling direction RD of the strip. The two rotations may be carried out in any order desired, but for the purpose of illustration the rotation about the MP axis will be considered as being made first. Starting with the strip **1** as shown in Fig. 5 the crystal B is rotated through some angle ϕ (after heating neck **4** to red heat to make it deformable) which will bring one direction of the crystal into the desired orientation. The crystal position after this rotation is shown in Fig. 6. The second rotation about an axis such as TU normal to the strip **1** is best shown in Figs. 3 and 4. In Fig. 3 the crystal B is shown as being removed from its desired direction about TU by an angular amount equal to θ. With the neck **4** at red heat the crystal B is rotated about TU as an axis to bring the crystal into the position shown in Fig. 4.

To take a specific example, suppose it is desired to reorient a crystal B′ as shown in Fig. 7 to the (110) [001] orientation, that is, with the (110) plane in the plane of the strip and with the [001] direction parallel to the rolling direction RD. In the explanation the [110] direction **5** which is perpendicular to the (110) plane, is

used in lieu of the latter and the normal 6 to the sample is used in place of the plane of the sample to simplify the drawing. The first step is to rotate the crystal about MP through angle ϕ so that the [110] direction 5 coincides with the normal 6 to the sample as shown in Fig. 8. This operation brings the (110) plane in the plane of the sample but the [001] direction may still be out of line with the rolling direction RD by some angular amount θ. To bring [001] into a position parallel to RD the crystal B' is rotated about the normal 6, which now coincides with the [110] direction 5, an amount θ to attain the condition shown in Fig. 9. In this final (110) [001] position the [110] direction is shown as being vertical, which means that the (110) plane lies in the plane of the strip and the [001] direction is parallel to the rolling direction RD.

Having obtained the desired orientation of the seed crystal, the sample is fed into a temperature gradient furnace maintained at a temperature which will support the growth of the seed crystal but which will not start the growth of differently oriented crystals and at such speed as is equal to the natural rate of growth of the crystal for the temperature.

In actual operation several specific silicon steels have been transformed into desired orientations. However, it is emphasized that the process described herein is not to be limited to specific compositions but includes all magnetic materials whether of face centered or body centered crystal structure, inasmuch as one skilled in the art, will be able using the teachings described herein, so to convert magnetic materials of other compositions into a form having a very high degree of preferred crystal orientation.

The single crystal thus produced may have a thickness ranging from about 0.005 to 0.050 inch. It is to be emphasized that crystal growth took place in a strip material already rolled to size. If the material had been rolled to a thickness of less than 0.005 inch a single crystal would not have emerged from the subsequent heat treatment. In order to confer the maximum possible degree of magnetic orientation to magnetic material having a thickness of less than 0.005 inch a single crystal strip about 0.005 inch in thickness prepared as described above is cold rolled to reduce its thickness by about 70 per cent. It is then recrystallized at a temperature of about 980° C. These last cold rolling and crystallizing steps may be taken in two or more stages starting with thicker single crystal material.

Magnetic material produced in accordance with the above described method is not a single crystal but has a preferred crystal orientation closely approaching that of a single crystal. A quantitative pole figure of the desired final product is illustrated in Fig. 10. If a final product possessing the most desirable preferred orientation is to be produced close control over the steps of the process must be maintained. This is particularly important with respect to step 2 dealing with orientation of the seed crystal and step 5 dealing with the recrystallization treatment.

The magnetic torque test illustrated in Fig. 11 emphasizes the importance of seed crystal orientation. Three test specimens, indicated as S1, S2, and S3 on the graph were given different orientations as follows: specimen S1 had a tilt of less than 3 degrees of the [001] direction up from the rolling direction; specimen S2 had a tilt of 13° from this direction; specimen S3 had a tilt of 25° from this direction. All three specimens had a deviation of [110] from the cross rolling direction of less than 1½°. From Fig. 11 it may be seen that specimen S1, which had the least tilt, was far superior to the other two and that specimen S3, which had a tilt of 25°, gave a very unsatisfactory test.

If deviating primary recrystallization grains and secondary recrystallization grains are to be avoided it is important that the final recrystallization temperature be kept below 1000° C. and preferably at a temperature of about 980° C. I have found that when primary recrystallization produces a high percentage of primaries in one preferred orientation and the remaining primaries in other orientations in such a way that a number of the latter have diameters 2 or 3 times the average, then the large primaries in deviating orientation grow into secondaries. If the recrystallization conditions favor the growth of deviating primary crystals and secondary crystals the quality of the final product is lower. Secondary, recrystallization proceeds at a low rate below 1000° C. and at a rapid rate near 1100° C. The induction period decreases and numbers of secondary crystals increases with rising temperatures. On the other hand, primary recrystallization occurs slowly below 600° C. and fairly rapidly near 700° C. Thus, a recrystallization temperature of about 980° C. produces optimum results. However, it is emphasized that lower recrystallization temperatures do not have an adverse effect on the quality of the final product and a temperature of 700° C. is satisfactory for the recrystallization step.

One material used in my process was a silicon steel of from 3.0 per cent to 3.5 per cent silicon content which was cold rolled to a final thickness in the range 0.005 to 0.025 inch by a two stage cold rolling process from an initial thickness of about 0.1 inch with an intermediate continuous anneal at 850° C. between stages. A group of crystals were grown as described above in one end of the piece and one of the best crystals selected by X-ray methods and isolated with a neck connecting it to the main strip as outlined. It was found that the matrix produced by the above treatment of such a steel would support the growth of crystals having orientations only near (110) [001] at a temperature of 900° C.–1000° C. when fed into the gradient temperature furnace at a rate of 0.2 to 2.0 inches per hour which also represents the rate of growth of the single crystal of preselected orientation.

The material undergoing treatment may be strained either by being pulled in tension or by being cold rolled prior to the transformation to a single crystal. A reduction in thickness in the range 2% to 6% is preferred for a cold rolling strain.

While the present invention has been described with reference to particular embodiments thereof, it will be understood that numerous modifications may be made by those skilled in the art without actually departing from the invention. Therefore, I aim the appended claims to cover all such equivalent variations as come within the true spirit and scope of the foregoing disclosure.

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

1. The method of producing thin oriented magnetic strips which comprises cold rolling a silicon steel strip of an initial thickness of about 0.005 to 0.025 inch by a two-stage process with an intermediate anneal at 850° C. between stages, heating one end of the cold rolled silicon steel strip to grow a number of crystals in said end, selecting one of said crystals as a seed crystal for further growth, cutting away areas from the end of said strip to leave only the selected crystal connected to the main portion of said strip by a neck portion of the silicon steel material, heating the neck portion and in successive operations bending the neck portion about an axis parallel to the plane of the strip and an axis perpendicular to the plane of the strip to bring the selected seed crystal into (110) [001] orientation with reference to the main portion of said strip, introducing the end of the strip including said seed crystal into a temperature gradient furnace to heat said strip to a temperature of 900° C.–100° C., moving said strip into said furnace at a rate equal to the rate of growth of said seed crystal, further cold rolling said strip to bring about a reduction in thickness of about 70 per cent, and recrystallizing said strip at a temperature between about 700° C. and 1000° C. in a reducing atmosphere.

2. The method of claim 1 wherein the final cold rolling and recrystallizing treatment is repeated.

3. The method of producing silicon steel magnetic strip material having a high degree of preferred magnetic orientation which comprises isolating a crystal at one end of a silicon steel strip, cutting away the crystals from said one end to leave said crystal connected by a thin neck to the remainder of said strip, orienting the crystallographic directions of the crystal by plastically deforming the neck at red heat until said crystal has a (110) [001] orientation with reference to the main portion of said strip, introducing the end of the strip including said seed crystal into a temperature gradient furnace to heat said strip to a temperature of 900° C.–1000° C., moving said strip into said furnace at a rate equal to the rate of growth of said seed crystal, cold rolling said strip to bring about a reduction in thickness of about 70 per cent, and recrystallizing said strip at a temperature be-

tween about 700° C. and 1000° C. in a reducing atmosphere.

4. The method of claim 3 in which the reducing atmosphere is hydrogen.

5. The method of producing silicon steel magnetic strip material having a high degree of preferred magnetic orientation which comprises isolating a single crystal at one end of a silicon steel strip by cutting away other crystals from said one end to leave said crystal connected by a thin neck to the remainder of said strip, orienting the crystallographic directions of the crystal by plastically deforming the neck at red heat until said crystal has a (100) [001] orientation with reference to the main portion of said strip, introducing the end of the strip including said seed crystal into a temperature gradient furnace to heat said strip to a temperature of 900° C.–1000° C., moving said strip into said furnace at a rate equal

to the rate of growth of said seed crystal, subjecting said strip to a treatment consisting of cold rolling said strip to reduce its thickness by at least 70 per cent followed by heating said strip to a recrystallizing temperature of about 980° C. in a reducing atmosphere, and repeating said treatment at least once.

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