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PROCESS FOR PRODUCING PREDETERMINED SECONDARY RECRYSTALLIZATION TEXTURES IN METALS

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This invention relates to a process for producing bodies of metallic material with a predetermined secondary recrystallization texture.

It is often desirable to produce metallic members having a crystalline texture which is obtained by secondary recrystallization thereof. Many bodies of metals when annealed at suitable temperatures can form by secondary recrystallization one or more grains having a predetermined orientation with respect to the surface. Some crystalline textures that may be produced by secondary recrystallization are much more desirable for certain purposes than are others. Thus, for example, in silicon steel sheets there may be produced a cube-on-face grain texture which is identified as the (100) [001] texture in Miller indices. An equally possible secondary recrystallization texture in silicon steel is the cube-on-edge or (110) [001] grain texture. A number of factors may cause the secondary recrystallization of the silicon steel so as to grow grains preferentially according to one of these textures or some other texture depending upon the treatment of the sheet of the silicon steel such cold rolling, surface films, composition of the steel, the composition of the atmosphere, annealing temperature, length of anneal, and other factors which have been found to influence the preferential growth of a particular secondary recrystallization texture. It would be desirable if there were available a technique for forcing or causing a particularly desired secondary recrystallization texture to be induced in a metallic member.

The terms "primary recrystallization" and "secondary recrystallization" are well known in the art. In connection with silicon iron alloys, these terms are described in the following two articles by C. G. Dunn and in the references listed at the end thereof: Acta Metallurgica, vol. 2, March 1954, pages 174–183, and Acta Metallurgica, vol. 2, No. 3, May 1954, pages 386–393. Briefly, primary recrystallization of a metal is the initial crystal texture that forms or grows out of the deformed grain texture of a body of metal, normally hot or cold worked. Thus, a cold rolled sheet of silicon-iron presents a more or less severely deformed grain texture such that upon subjecting the cold rolled sheet to annealing, first stress relief occurs rapidly, usually with little change in grain texture, then concurrently with such stress relief or soon thereafter, a recrystallization occurs in which a different grain texture, designated as the primary recrystallized texture develops in which certain grains form by absorbing the deformed grain texture. This primary recrystallized texture differs both in grain size and orientation of the grains from the deformed grain texture of the sheet being subjected to anneal. For silicon steel primary recrystallization may begin at temperatures of about 500° C. and higher, the rate of recrystallization being dependent on the temperature as well as the severity of the preceding cold rolling step.

If the temperature of the annealing of the silicon steel is high enough, usually above 900° C., and ordinarily above 1000° C., the initially formed primary recrystallization texture will begin to exhibit growth of grains of a still different orientation than the primary grains, the rate of such growth being dependent upon the temperature of

the annealing. This latter texture is designated as secondary recrystallization. In some cases secondary recrystallization may be initiated before primary recrystallization of the entire sheet or member is completed. The secondary grains are almost invariably much greater in size than the primary grains.

The extent of secondary recrystallization is dependent on many factors. In some instances, the primary recrystallization texture forms readily but only a part of the primary recrystallized texture will convert to or form secondary grains. Consequently, it is common to find a sheet with a relatively small number of secondary grains, relatively large in size, in a surrounding matrix or area composed of many fine primary recrystallized grains. The sheet volume may comprise as little as 10% to 20% secondary grains to as much as 80% or more. Sometimes by increasing the annealing temperatures the secondary grain texture may be caused to occur over a larger portion of the volume of the sheet.

It is well known that secondary recrystallization is not only temperature and time dependent but also is influenced by factors such as composition of the metal or alloy being annealed, its previous rolling and annealing history and other factors. It would be desirable to have available a process for so controlling secondary recrystallization that a particular crystallization can be induced in a member and carried through to substantially complete conversion of the grain texture of the sheet regardless of its composition, previous history or other factors. This is particularly desirable in magnetic materials such as nickel-iron, silicon-iron and aluminum-iron alloys. However, it will be evident to the metallurgist that numerous other metals may be advantageously processed if there were some means of predetermining and controlling the texture that may be imparted thereto by annealing carried out to secure secondary recrystallization.

It is possible, for example, to produce in heavily cold reduced silicon-iron alloys containing from 2% to 5% silicon a cube texture by secondary recrystallization. For this purpose one process comprises cold rolling in one or preferably a plurality of steps with reductions amounting to from 40 to 90% per pass and, if required, with intermediate anneals at temperatures of from about 750° C. to 950° C. Following the desired cold reduction to produce a sheet of desired final gauge the sheet is subjected to a final anneal which results in secondary recrystallization. This final anneal is carried out at temperatures of from about 1100 to 1350° C. in a very pure atmosphere, for example in hydrogen which is extremely low in oxygen, preferably of a dew point of below −40° C., or in a vacuum which has an absolute pressure of 10^{-2} mm. Hg and preferably 10^{-3} mm. Hg and less. Such process has given in many instances the desired cube-on-face recrystallization texture. From experience, it has been found that the desired cube-on-face secondary recrystallization is not always certain to result because due to, for example, impurities in the metal these may prevent the growth of a desired secondary recrystallization texture. In some instances, a failure to observe some of the cold rolling and intermediate requirements may render it very diffcult to secure an appreciable amount of secondary recrystallization. These problems become progressively greater as the sheets or strips increase in thickness, for example, sheets of a thickness of more than 0.35 mm. are often difficult to anneal so as to obtain any high volumetric proportion of cube-on-face secondary recrystallization texture.

The present invention relates to a novel method for effecting a desired secondary recrystallization in metals which exist in a state wherein the metal does not lend itself to the desired secondary recrystallization texture

or is only slightly susceptible to such secondary crystallization growth. A member is said to have little susceptibility to desired secondary recrystallization when an anneal carried out at a temperature above the recrystallization temperature of the respective metal member results in a secondary recrystallization of less than about 80%, for example less than 20%, of the sheet or strip volume. The present invention will be suitable for obtaining any desired proportion of secondary recrystallization of the sheet or strip volume providing the metal is inherently capable of assuming the desired secondary recrystallization.

The materials to which the present invention may be applied are either cold worked, for example, cold rolled, sheets, strips, plates, or the like or even primary recrystallized sheets, strips, plates, or the like of any desired metal or alloy. Of course, the invention will not be suitable for application to a metal member which is already in a secondary recrystallized condition. Any metal capable of secondary recrystallization by heating to some predetermined temperature can be processed in accordance herewith. Particularly suitable members comprise any rolled metal or alloy which after the rolling operation is capable of assuming a secondary recrystallized texture.

The object of this invention is to provide a process for causing a metal member to attain a desired secondary recrystallization texture by bringing into intimate contact with the surfaces of the metal member a stimulating metal member which will readily undergo secondary recrystallization and heating the assembly to a temperature such that, first, the two members will weld at least partially along the contacting surfaces thereof and thereafter the stimulating member will undergo secondary recrystallization which secondary recrystallization texture will convert the first metal member to a similar crystalline texture by continuation of the annealing.

A further object of the invention is to provide a process in which a cold rolled sheet of metal capable of undergoing secondary recrystallization on annealing by applying in intimate contact at all portions of one surface at which secondary recrystallization is desired an inducing metal sheet capable of readily undergoing the desired secondary recrystallization, the inducing sheet being of a thickness of at least equal to the thickness of the cold rolled sheet, maintaining the two sheets in intimate contact and subjecting them to an annealing temperature such that the inducing sheet will undergo secondary recrystallization and thereby induces a similar secondary recrystallization of the cold rolled sheet.

A still further object of the invention is to place in intimate contact two metal members over their surface areas so intimately that upon being subjected to annealing at least partial welding will take place, the metal members having a similar crystal structure at least at annealing temperatures, one of the metal members being more readily converted on annealing to a predetermined secondary recrystallization texture which secondary recrystallization texture will thereby be induced in the other metal member.

Other objects of the invention will in part, be obvious and will in part, appear hereinafter.

In accordance with the present invention a first metal member can be caused to attain a desired secondary recrystallization texture in a predetermined amount, providing that the secondary recrystallization texture is inherently possible for this metal upon annealing, by applying thereto an inducing member comprising a metal which will readily undergo secondary recrystallization to convert at least 80% of its volume to a desired secondary recrystallization texture, the surfaces of the two members being in intimate contact over predetermined portions of the first metal member which it is desired to cause to undergo the desired secondary recrystallization, and then subjecting the composite structure to heating to annealing temperatures whereby there occurs at least a partial welding of the two members at their contacting surfaces and the inducing member will undergo secondary recrystallization whereby it causes a similar secondary recrystallization texture to occur in the first member.

In some instances the second or inducing member may already have a desired secondary recrystallized texture before it is jointed to the first member which latter may be either in a cold worked or primary recrystallized state. The two members are then placed in intimate contact and annealed so that the desired partial or complete welding of the two members takes place and then the structure is subjected to a temperature high enough so that secondary recrystallization of the first member is possible and under these conditions the secondary recrystallization texture already present in the inducing member will be imparted to the first member.

It should be recognized that for successful practice of the invention, both members must have the same crystal structure at the annealing temperature. For example, both metals must, at least at the annealing temperature, be cubic body centered, or face centered, or the like. It is well known that in some cases metals may have different structures at room temperature than they have at annealing temperatures. However, if the structures are similar at the annealing temperature then the inducing metal will induce the desired secondary recrystallization texture in the member being treated.

The desired secondary recrystallization annealing preferably takes place in an atmosphere which is neutral, or reducing, with respect to the metals being treated. For example, hydrogen, preferably dry hydrogen of a dew point of less than −30° C., or an inert gas such as argon or helium, or a vacuum at an absolute pressure of not greater than 10^{-1} mm. Hg, may be employed for the annealing atmosphere.

As examples of metal members that may be treated in accordance with the invention, it is preferable to employ cold worked or cold rolled sheets or plates. However, hot rolled members which may be partially cold rolled to produce a desired surface finish or even cast members may be treated in accordance with the invention. Aluminum and its alloys, various ferrous metals such as carbon steel, iron-chromium, iron-nickel, and iron-silicon alloys, nickel and cobalt and alloys thereof, as well as copper and copper base alloys are examples of metals that may be processed in accordance with the present invention.

It is necessary that the surfaces of the sheet to be treated and the inducing sheet be placed in closely conforming contact so that welding may be able to occur between the surfaces. Therefore the contacting surfaces should be clean in order to obtain a metallurgical contact therebetween. Furthermore, the sheets should be so prepared that they conform or can be made to conform to each other whereby adequate metallurgical bonding or welding may be caused to occur. For sheets of metal it is possible to place the two sheets together and cold roll them several times to effect both cold reduction and the required intimate contact between the surfaces. Such doubled, cold rolled sheets may be annealed at the proper secondary recrystallization temperatures and obtain a partial or complete welding therebetween.

In connection with the annealing of the sheet to be secondarily recrystallized and the inducing sheet it may be necessary to conduct the anneal so that there will be a desired crystal configuration equalization between the metals. This may be necessary, for example, to insure that in both sheet components there will be the same crystalline structure at the annealing temperature. For instance, if a sheet consisting of a silicon-iron alloy is annealed together with a sheet consisting of pure iron, then at a high annealing temperature the silicon-iron would exist in the alpha state and the pure iron sheet would exist in the gamma state. However, by performing the anneal so that part of the silicon will diffuse from the silicon-

iron sheet into the pure iron sheet, the latter sheet component will still be present as essentially pure iron but it can be caused to assume the alpha state. Consequently, the annealing requirements should be so selected that there is a desired balancing of the concentration of components so that when the anneal is performed that the composition or concentration unbalance which tends to favor different crystal modifications will only be slight or will not take place at all. This can be attained, for example, by making the anneal period as short as possible in order to prevent the conversion of the phase of the metal to an undesirable crystal state in either of the components.

The following examples are illustrative of the practice of the invention, particularly for enabling cube texture grain growth in silicon-iron alloys having from 1% to 7% silicon.

Example I

A sheet of silicon-iron alloy containing 2.9% silicon and of a thickness of 0.24 mm. was produced by cold rolling. This sheet was shown to be capable of secondary recrystallization on annealing to produce substantially a major proportion of its volume of crystals having the cube-on-edge or Goss orientation, namely, the (110) [001] texture and the sheet also was capable of secondary recrystallization into a cube orientation texture, that is, (100) [001] orientation, depending on the annealing conditions. There was placed on this sheet of a thickness of 0.24 mm. a sheet of pure annealed iron of a thickness of 0.1 mm. This latter sheet had a primary recrystallized fine grain texture. These sheets were put into close conformation of the surfaces to each other. After annealing the composite structure for 20 hours at 1180° C. in a stream of dry hydrogen (dew point below —40° C.) it was found that many crystals had grown from the silicon-iron strip through the pure iron sheet. These crystals included large crystals having the cube texture. An analysis had indicated that silicon had diffused from the silicon-iron strip into the pure iron strip.

Example II

Upon a strip of the same silicon-iron alloy as in Example I, the strip being of a thickness of 0.24 mm., there was placed a strip of a thickness of 0.3 mm., the latter strip consisting of a silicon-iron alloy containing 2.82% of silicon. The latter strip had been heavily cold rolled but would not secondarily recrystallize to any appreciable extent under selected annealing conditions. The strips were so disposed upon each other that a substantial portion of each overlapped the other, but each strip protruded beyond the other at each end. The assembly of both strips was then annealed for 20 hours at a temperature of 1180° C. in a stream of dry hydrogen gas. After the anneal it was found that the protruding end of the strip of a thickness of 0.3 mm. was not secondarily recrystallized to any appreciable extent. However, in the common zone where the two strips did overlap they had welded together and a very large proportion of the volume of the secondary crystals developed in the strip of a thickness of 0.24 mm. had grown into and through the upper sheet which had the thickness of 0.3 mm. At the conclusion of the annealing there was obtained a composite sheet having a total thickness of 0.54 mm. characterized by substantially complete secondary recrystallization except for the protruding end of the strip of a thickness of 0.3 mm. This example illustrates the fact that it is possible to produce a desired secondary recrystallization in sheets of great thickness. As is well known, it becomes increasingly more difficult to attain secondary recrystallization as the thickness of the material is increased.

Example III

There was placed upon a strip of a thickness of 0.24 mm. consisting of the 2.9% silicon-iron alloy of Example I, a strip of a thickness of 0.1 mm. said latter strip consisting of a silicon-iron alloy having 3.12% silicon. This last-mentioned strip had been heavily cold rolled. As in Example II, these two strips were so disposed upon each other that a free end of each protruded beyond the portion where they overlapped each other. The strips were then compacted so that their surfaces conformed closely to each other. The assembly was then placed in an annealing furnace where it was annealed for 20 hours at a temperature of 1180° C. in an atmosphere of dry hydrogen. After the anneal, the exposed portion of the strip of a thickness of 0.1 mm. had secondarily recrystallized up to 70% of its volume to produce crystals having cube faces parallel within approximately 5° to the plane of the surface of the sheet, while the remainder of the volume of this exposed portion of the sheet comprised fine grains of a size of an average diameter of 1 millimeter or less. The overlapping portion of the two strips where they had welded together exhibited only a completely recrystallized secondary structure and no appreciable amount of the fine grained texture which was present up to 30% of the volume of the 0.1 mm. strip. In other tests as much as 50% of the protruding end of the strip of a thickness of 0.1 mm. exhibited 50% of the fine grained structure which was primary recrystallized texture material.

Example IV

Upon the 0.24 mm. strip of Example I, there was placed a second cold rolled strip comprising a silicon-iron alloy having 3.12% silicon, the latter strip having a thickness of 0.3 mm. Tests had indicated that the strip of a thickness of 0.3 mm. was extremely difficult to secondarily recrystallize under any reasonable annealing condition. However, after 20 hours of annealing at 1180° C. in a stream of dry hydrogen, substantially complete secondary recrystallization was observed throughout the composite member composed of the two strips where they were in contact with each other. An analysis of the secondary crystals in this composite member indicated that the dodecahedral planes (110) and octahedral planes (111) were substantially parallel to the surface of the sheet or the plane of rolling.

Example V

Two silicon-iron strips which had been previously secondarily recrystallized and had different crystal orientations were placed upon one another and the surfaces caused to closely conform to each other. These strips were then annealed as in Example I until they had welded together. After the annealed strips were examined, it was found that they had not affected the crystal structure of each other. This indicates that once secondary recrystallization has been obtained in a sheet it normally will not be influenced by another sheet that has secondary recrystallization texture therein.

The effectiveness of the influencing sheet upon the sheet to be converted to a predetermined secondary recrystallized texture depends not only upon the annealing condition such as temperature, time and atmosphere but also on the relative thickness of the superimposed strips or sheets. As a general rule the thickness of the sheet or strip which will not readily secondary recrystallize or at most has only a small amount of secondary recrystallization should be of a thickness equivalent to that of the inducing sheet or possibly smaller.

It will be understood that after the annealing there will be obtained a single substantially welded sheet product. These sheets are not intended to be separated after annealing and as a rule it is impractical to separate them because they are welded. Consequently, the process will enable the attainment of a desired secondary recrystallization structure in metallic materials which do not lend themselves readily to secondary recrystallization. Also it makes it possible to produce members having a secondary recrystallization structure combined with a desired texture, of a thickness which previously has never

been secondarily recrystallized or it has been difficult to cause it to occur. Furthermore, where a sheet of material which, after having been finished is discovered not to undergo secondary recrystallization, desired secondary recrystallization may be stimulated in accordance with the present invention, and therefore, the sheet may be made available for its desired use.

The stimulating sheet will ordinarily be at least approximately equal to the thickness of the other sheet, though in some cases it may be somewhat thinner than the sheet in which secondary recrystallization is to be induced.

This application is based upon German patent application V–16,699 VI/40d, filed June 12, 1959, and assigned to the assignee of the present invention.

It will be understood that the above description is only illustrative and not limiting of the invention.

I claim as my invention:

1. In the process of causing a predetermined secondary recrystallization texture in a first sheet of a metal which can attain such secondary recrystallization texture but has not been previously subjected to secondary recrystallization, the steps comprising disposing said first metal sheet in intimate contact of selected portions of its surface with a surface of a stimulating sheet of a metal which at the annealing temperatures necessary to effect secondary recrystallization has the same crystal structure as the said first metal sheet and having a crystal structure that will yield the predetermined secondary crystal structure during secondary recrystallization, the contacting surfaces of the sheets being clean, the stimulating sheet readily attaining a given secondary recrystallization texture at the annealing temperature, and heating the assembled sheets to annealing temperatures, the temperature, the cleanliness, and the intimate contact between the surfaces producing at least a partial welding of the surfaces, and continuing the annealing until secondary recrystallization grain growth of the stimulating sheet has occurred and has induced a corresponding secondary recrystallization texture in the first sheet.

2. In the process for producing a secondary recrystallization grain texture in a metal body in which it is relatively difficult to produce such secondary recrystallization grain texture, the steps comprising disposing a stimulating metal member in intimate contact over a portion of the surface of the metal body, at least the contacting portions of the surfaces being clean, the stimulating metal member having a crystal structure such that it is capable of readily undergoing substantially complete secondary recrystallization to yield a specified secondary crystal structure when heated to a secondary recrystallization annealing temperature, the metal member and the metal body having the same crystal structure at the annealing temperature, heating the intimately joined metal member and metal body to a temperature to effect at least partial welding thereof at the said portion of the surface of the metal body, and continuing heating to the said annealing temperature until the stimulating metal member undergoes secondary recrystallization to yield the specified crystal structure and thereby induces a corresponding secondary recrystallization grain growth of the metal body at the said portion of the surface thereof.

3. In the process for producing secondary cube grain recrystallization grain texture in a first metal sheet in which it is relatively difficult to cause secondary recrystallization grain growth to a desired volumetric extent, the steps comprising disposing a second stimulating metal sheet in intimate contact with a selected portion of the first sheet, the contacting surfaces of the sheets being clean, the stimulating metal sheet having a crystal structure so that it is capable of readily undergoing substantially complete secondary recrystallization to yield a cube texture when heated to a secondary recrystallization annealing temperature, the two metal sheets having the same crystal structure at the annealing temperature, the thickness of the first sheet being not greatly in excess of the thickness of the stimulating sheet, heating the intimately joined metal sheets to a temperature equal to at least the said annealing temperature to effect at least a partial welding of the two sheets at their intimately disposed portions and continuing the heating until the stimulating sheet undergoes secondary recrystallization to yield the cube texture and thereby induces a corresponding secondary recrystallization cube texture grain growth in the first sheet.

4. The process of claim 3, in which the sheets comprise a silicon-iron alloy having from 1% to 7% of silicon, and the annealing is effected in a reducing atmosphere.

5. The process of claim 3, in which the sheets comprise a silicon iron alloy having from 1% to 7% of silicon, and the inducing sheet has over 80% of its volume comprised of cube-on-face grain texture.

6. In the process for producing secondary recrystallization grain texture in a first metal sheet in which it is relatively difficult to cause secondary recrystallization grain growth to a desired volumetric extent, the steps comprising disposing a second stimulating metal sheet in intimate contact with a selected portion of the first sheet, the contacting surfaces of the sheets being clean, the stimulating metal sheet having a desired secondary recrystallization grain texture, the two sheets having the same crystal structure when heated to an annealing temperature effective to produce some secondary recrystallization in the first sheet, the thickness of the first sheet being not greatly in excess of that of the stimulating sheet, heating the intimately joined sheets to a temperature equal to at least the said annealing temperature to effect at least a partial welding of the two sheets at their intimately disposed portions and continuing the heating whereby the stimulating sheet induces a corresponding secondary recrystallization grain growth in the first sheet.

7. In the process for producing secondary recrystallization grain texture in a first metal sheet in which it is relatively difficult to cause secondary recrystallization grain growth to a desired volumetric extent, the steps comprising disposing a second stimulating metal sheet in intimate contact with a selected portion of the first sheet, the contacting portions of the sheets being clean, the stimulating metal sheet having a crystal structure so that it is capable of readily undergoing substantially complete secondary recrystallization to yield a given secondary crystal structure when heated to a secondary recrystallization annealing temperature, the two metal sheets having the same crystal structure at the annealing temperature, the thickness of the first sheet being not greatly in excess of the thickness of the stimulating sheet, the two joined sheets being cold rolled at least once in order to cause the surfaces to conform more closely, heating the intimately joined metal sheets to a temperature equal to at least the said annealing temperature to effect at least a partial welding of the two sheets at their intimately disposed portions and continuing the heating until the stimulating sheet undergoes secondary recrystallization to yield the specified secondary crystal structure and thereby induces a corresponding secondary recrystallization grain growth in the first sheet.

8. In the process for producing secondary recrystallization grain texture in a first metal sheet in which it is relatively difficult to cause secondary recrystallization grain growth to a desired volumetric extent, the steps comprising disposing a second stimulating metal sheet in intimate contact with a selected portion of the first sheet, the contacting surface portions being clean, the stimulating metal sheet having a crystal structure so that it is capable of readily undergoing substantially complete secondary recrystallization to yield a desired secondary crystal structure when heated to a secondary recrystallization annealing temperature, the two metal sheets having the same crystal structure at the annealing tempera-

ture, the thickness of the first sheet being not greatly in excess of the thickness of the stimulating sheet, the sheets having differences in composition by reason of a readily diffusable component being present in one sheet, heating the intimately joined metal sheets to a temperature equal to at least the said annealing temperature to effect at least a partial welding of the two sheets at their intimately disposed portions and continuing the heating for a period of time to cause a diffusion of the readily diffusable component from the one sheet into the other sheet whereby to produce a compensation of the component between the two sheets at least at the welded

surfaces, the annealing being continued until the stimulating sheet undergoes secondary recrystallization to yield the desired secondary crystal structure, and thereby induces a corresponding secondary recrystallization grain growth in the first sheet.

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