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3,676,227

PROCESS FOR PRODUCING SINGLE ORIENTED SILICON STEEL PLATES LOW IN THE IRON LOSS

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ABSTRACT OF THE DISCLOSURE

A process for producing a single oriented silicon steel plate or particularly a single oriented silicon steel plate having a high magnetic induction produced by adding a small amount of Al in the molten steel characterized by that in heating steel plate it is painted with an annealing separating agent, to which boron or boron compound is added in a prescribed amount, and then finally annealed, whereby the iron loss can be remarkably reduced.

BACKGROUND OF THE INVENTION

Field of invention

The present invention is to provide a method for reducing an iron loss of a single oriented silicon steel plate or particularly a single oriented silicon steel plate produced by adding a small amount of Al to the molten steel. A single oriented silicon steel plate is a magnetic steel plate in which the crystals forming the steel have an orientation represented as (110) [001] in Miller's index and which is easily magnetized in the direction of the rolling of the plate. The magnetic characteristics required of a single oriented silicon steel plate are a high magnetic induction and a low iron loss characteristic. The iron loss is an important characteristic influencing the efficiency of a transformer, generator or motor made of such a single oriented silicon steel plate. The purpose of the present invention is to reduce this iron loss.

Description of the prior art

Factors which influence the above-mentioned iron loss include thickness, specific resistance, impurity, orientation and crystal granularity of the iron plate. Adjustable among these factors are the impurity and orientation. As regards the impurity, the single oriented silicon steel plate produced for commercial uses today is usually so well purified that only a very small amount of the impurity is contained in the final product. As regards the orientation, needless to say, efforts are being made so that the crystals may be well singly oriented. The influence of the crystal granularity on the iron loss is complicated. As is well known, when the iron loss is analyzed, it is found that the iron loss consists of two factors, i.e. an eddy current loss and a hysteresis loss. As the crystal granularity becomes larger, the hysteresis loss is reduced. On the other hand, the eddy current loss tends to increase as the crystal grain becomes larger. Particularly, when the crystal grain reaches a certain size, the eddy current loss increases quickly to exceed the reduction of the hysteresis loss, whereby the total iron loss increases. Therefore, in order to obtain a single oriented silicon steel plate having a low iron loss, it is necessary to make the crystal grain properly small.

As is well known, a single oriented silicon steel plate is made by utilizing a secondary recrystallization phenomenon. The mechanism for the generation of the sec-

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ondary recrystals is that, when a material treated by a combination of a cold-rolling and annealing is finally annealed at a high temperature, grains having a specific orientation, that is, an orientation of (110) [001] or slight deviations from this orientation among primary recrystal grains having various crystal orientations, quickly grow, thereby encroaching on the matrix until the steel plate is occupied substantially completely by only these grains. In this case, therefore, it is not possible to utilize conventional measures usually adopted as effective to regulate the crystal granularity such as the regulation of the cold-rolling reduction rate, the annealing temperature or the addition of an impurity. Furthermore, there has been thus far reported no effective method of adjusting the secondary recrystal granularity. It is a known fact that for the purpose of inducing the secondary recrystallization it is necessary to add an impurity such as MnS, MnSe, AlN or VN to the steel so that a deposited dispersion phase may be formed. The role of such an impurity is to inhibit the growth of the primary recrystal grains of the matrix so that the matrix may be kept microgranular and may be easily encroached by the secondary recrystal grains. Therefore, it is not effective to the inhibition of the growth of the secondary recrystal grains. Further, after the secondary recrystals have been formed, these deposits are dissociated and solid-dissolved in the steel or are removed from the surface of the steel plate by diffusion.

Today, the above described view on the eddy current loss is well known. However, the present inventors have discovered that there is still a factor which performs an important action on the eddy current loss of a steel plate having a high magnetic induction as is disclosed, for example, in the specifications of U.S. Pat. 3,287,183. This factor is a glassy film formed on the surface of the steel plate by the reaction of the steel plate with an annealing separating agent when the steel plate is painted with the annealing separating agent and is finally annealed at a high temperature.

Such a glassy film produced by the reaction of a steel plate with an annealing separating agent in the final annealing is utilized extensively as an insulating film for single oriented silicon steel plates. Another effect of this glassy film is the reduction of magnetostriction. That is to say, due to the difference in the thermal expansion rate between the glassy film and steel, the glassy film gives a tension to the steel plate, whereby magnetostriction is reduced.

SUMMARY OF THE INVENTION

With the above in mind, it is an object of the present invention to produce a single oriented silicon steel plate, or particularly a single oriented silicon steel plate having a high magnetic induction, which is characterized by having a low iron loss by making secondary recrystals of the steel plate properly small and by forming a stable glassy film uniformly on the surface of the steel plate.

Other objects will be made clear by the following description.

A concrete method for obtaining the above-mentioned object of the present invention is to paint a single oriented silicon steel plate, particularly a single oriented silicon steel plate having a high magnetic induction, with an annealing separating agent, to which boron or a boron compound is added in a proper amount, when subjecting the said steel plate to final annealing. The amount of boron or a boron compound to be added is 0.01 to 1.0% by weight of the annealing separating agent. When the steel plate is painted with such an annealing separating agent containing boron or a boron compound as above-mentioned and is finally annealed, a single oriented silicon steel plate,

particularly a single oriented silicon steel plate having a high magnetic induction, will display a very low iron loss value.

DESCRIPTION OF THE PREFERRED EMBODIMENT

In the present invention the manner by which boron or a boron compound makes secondary recrystal grains small has not been perfectly theoretically clarified. However, it is believed that boron will diffuse into the steel from the surface during the final annealing and form a boride in the steel and that, even during annealing at a high temperature, this boride will not be decomposed and will prevent the growth of secondary recrystal grains. The secondary recrystal grains are large in size and are inherently difficult to grow due to the small orientation therebetween. It is believed to be due to this difficulty that the growth of the recrystals is prevented even by the slightest amount of impurity.

As to the effect of the method of the present invention in making the crystal granularity of the product fine, it is possible to obtain a product having a granularity smaller than any conventional products according to A.S.T.M. by adding 0.2% boron. Examples thereof are shown in the following:

Amount of addition of boron (percent):	A.S.T.M. No. (IX)
0.2 -----	5
0.4 -----	6.5
0.6 -----	9
0.8 -----	11

Further, the boron acts to improve and stabilize the properties of a film in that the remaining boron, other than that which has diffused into the steel, participates in the formation of a glassy film. That is, by the addition of boron, the interlayer resistance of the glassy film is approximately doubled, gas marks and the like vanish, fluctuations in the quality of the film due to fluctuations in the properties of the annealing separating agents such as MgO are prevented, and in addition thereto a thin adhering uniform film is formed over the entire surface. Thus, it is clear that boron acts to reduce iron loss by controlling the granularity of the secondary recrystals and by the formation of a high quality glassy film as above-mentioned. At any rate, it has been evidenced that, when a fixed amount of boron or a boron compound is added to the annealing separating agent during finishing annealing, a single oriented silicon sheet plate having a low iron loss can be obtained. The silicon steel material, which is the starting material of the present invention, may comprise a steel ingot made by solidifying by any casting method a molten steel prepared by a known steel making means such as, for example, an open-hearth furnace, an electric furnace or a converter or by any known melting means such as, for example, a high frequency electric furnace or a vacuum melting furnace. A slab-formed steel ingot obtained by a continuous casting method may also be used as a starting material of the present invention. The atmosphere in the case of the casting is usually air but may be a vacuum or inert gas. In short, the ingot in the present invention may be prepared by any steel making, melting or casting method.

For the oriented silicon steel plate to which the present invention is applied, it is desirable that the composition of the ingot contains, for example, less than 4.0% silicon, 0.010 to 0.065% acid-soluble Al, and less than 0.085% C. The ingot which is made into a slab by blooming after being cast or which is cast into a slab is made into a hot-rolled coil by continuous hot-rolling. The hot-rolled coil is treated by combined steps of cold-rolling and annealing. However, it is necessary that the treating steps and conditions should be those for obtaining single oriented silicon steel plates having a high magnetic induction.

That is, the final plate thickness is made by one or more

cold-rollings. In the case of a single cold-rolling, the cold-rolling reduction rate is made 81 to 95%, and in the case of two or more cold-rollings the final cold-rolling reduction rate is made 81 to 95%, after intermediate annealings are carried out one or more times. In either case, the plate is to be annealed at a temperature range of 1,000 to 1,200° C. for 30 seconds to 10 minutes after the hot-rolling or between the cold-rollings, so that AlN may be deposited. After this annealing for deposition, if necessary, the plate may be quenched from the temperature range of 750 to 950° C. down to 400° C. during 2 to 200 seconds depending on the contents of C and Si.

The steel plate made to be of the final thickness by the final cold-rolling should be made to have a carbon content of less than 0.005% by decarbonizing annealing. The surface of the steel plate after the decarburization is painted with an annealing separating agent to prevent the steel plate from sticking due to burning in the finishing annealing. Boron or a boron compound, which is an additive for obtaining a low iron loss as a characteristic of the present invention, is mixed into this separating agent to be applied onto the surface of the steel plate. The annealing separating agent may be a composition including any one or a combination of any of MgO, CaO, Al_2O_3 and TiO_3 .

Boron or a boron compound is added in the form of a powder or an aqueous solution to the annealing separating agent. With the addition of less than 0.01% boron to the annealing separating agent, no effect can be recognized. On the other hand, with the addition of more than 1% boron, the development of secondary recrystals is influenced and a single oriented silicon steel plate having a high magnetic induction can no longer be obtained. Therefore, the preferable amount of boron is from 0.02 to 0.5%. If 5 kg. of the annealing separating agent per ton of steel are to be used, the amount of boron required is surprisingly only 0.001 to 0.0025% by weight, and a sufficient effect can be obtained with this amount.

The final annealing should be carried out at temperature and for time sufficient for (100) 001 secondary recrystals to develop and for impurities to vanish due to the purification and annealing. For this purpose, it is necessary to anneal the plate above 1,000° C. for more than 5 hours in an atmosphere of hydrogen or nitrogen.

The present invention has been explained primarily with reference to oriented silicon steel plate containing Al. However, the present invention is not limited to such plates, but rather includes all single oriented magnetic steel plates.

EXAMPLE 1

A plate made by blooming and hot-rolling a silicon steel ingot containing 0.047% C., 2.80% Si, 0.030% Al and 0.030% S was annealed at 1,130° C., pickled, cold-rolled to be of a final plate thickness of 0.35 mm. and then annealed to be decarburized in a hydrogen atmosphere at 850° C. for 3 minutes. When this plate was painted with an annealing separating agent (MgO), into which 0.01 to 0.7% by weight boron had been added in the form of a powdery ferroboration of less than 200 meshes, and was finally annealed at 1,200° C., the results as shown below were obtained.

Amount of addition of boron into the annealing separating agent (percent)	Iron loss (w./kg.)		Iron loss improvement value (w./kg.)		Magnetic induction B_{10} (K.G.)
	W15/50	W17/50	W15/50	W17/50	
0 (conventional method)...	0.96	1.25	-----	-----	19.47
0.05-----	0.90	1.18	0.06	0.07	19.42
0.1-----	0.88	1.16	0.08	0.09	19.37
0.2-----	0.85	1.13	0.11	0.12	19.36

It is evident from these results that, when an annealing separating agent containing boron in the range of the present invention is used, there can be obtained a steel plate having a lower iron loss than any conventional

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steel plate painted with an annealing separating agent containing no boron.

EXAMPLE 2

Two plates of final plate thicknesses of 0.35 and 0.23 mm., respectively, were made by rolling a silicon steel ingot having a composition of 0.04% C., 2.8% Si, 0.034% Al and 0.027% S, the rest being Fe, by the same steps and conditions as shown in Example 1, and were annealed to be decarburized. Then, they were painted with an annealing separating agent (MgO) in the form of a slurry into which 0.2% by weight boron had been added in the form of borax, boric acid or single boron, and were finally annealed. The results as compared with the results of the conventional method are as follows, and it is evident that the steel plates made by the method of the present invention have lower iron losses.

Plate thickness (mm.)	Additives into the annealing separating agent	Iron loss		Iron loss improvement value		Magnetic induction B ₁₀
		W _{15/50}	W _{17/50}	W _{15/50}	W _{17/50}	
0.35	None (conventional method).	0.95	1.24	-----	-----	19.51
	Borax.....	0.87	1.15	0.08	0.09	19.49
	Boric acid.....	0.85	1.12	0.10	0.12	19.49
	Single boron.....	0.85	1.13	0.10	0.11	19.47
	None (conventional method).	0.77	1.08	-----	-----	19.36
0.23	Borax.....	0.68	0.96	0.09	0.12	19.32
	Boric acid.....	0.66	0.94	0.11	0.14	19.34
	Single boron.....	0.67	0.94	0.10	0.14	19.33

What is claimed is:

1. In a process for producing a single oriented silicon steel plate having high magnetic induction and low iron loss by hot-rolling a slab containing less than 0.085% by weight C., less than 4.0% by weight Si and 0.010 to 0.065% by weight acid-soluble aluminum, subjecting the thus hot-rolled steel plate to a single cold-rolling with a reduction rate of 81 to 95% or to two or more cold-rollings with intermediate annealings between them, in which the final cold-rolling rate is made 81 to 95% to make the final thickness of the steel plate, and in either case, annealing the plate at a temperature range of from 1000° to 1200° C. for from 30 seconds to 10 minutes

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after the hot-rolling, or between the cold-rollings, so that AlN may be precipitated in the steel plate prior to the final cold-rolling, then subjecting the cold-rolled steel plate having the final gauge to a decarburization annealing to reduce the carbon content of the steel to the value below 0.005% by weight, and thereupon to a final annealing at a temperature above 1000° C., the improvement comprising the steps of:

(a) coating the surface of said steel plate with an annealing separating agent consisting essentially of at least

(1) one compound selected from the group consisting of MgO, CaO, Al₂O₃ and TiO₂; and

(2) from .01 to 1.0% by weight, based upon the weight of said annealing separating agent, of a material selected from the group consisting of boron and a boron compound;

(b) drying the thus coated steel plate prior to a final annealing; and

(c) then subjecting said steel plate to a final annealing.

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