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(54) **A method for making grain-oriented silicon steel sheet having excellent magnetic properties**

Verfahren zur Herstellung von kornorientierten Siliziumstahlblechen mit ausgezeichneten magnetischen Eigenschaften

Procédé de production de tôles d'acier au silicium à grains orientés et ayant d'excellentes propriétés magnétiques

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DescriptionBACKGROUND OF THE INVENTION5 Field of the Invention

[0001] The present invention relates to a method for making a grain-oriented silicon steel sheet having excellent magnetic properties that remain consistent between different production lots and within individual sheets.

10 Description of the Related Art

[0002] Grain-oriented silicon steel sheets are mainly used as iron core materials for transformers and other electric devices. Required magnetic properties of iron core materials include high magnetic induction at a magnetic field of 800 A/m (B_8 , in units T); low core loss, i.e., low alternating current core loss at 50 Hz in 1.7 T of the maximum magnetic induction ($W_{17/50}$, in units W/kg); and the like.

[0003] Recent trends have required higher magnetic induction ($B_8 \geq 1.92$) in order to reduce core weight and noise. Furthermore, to improve fabrication efficiency and yield in large size transformers, homogeneous material characteristics are needed.

[0004] A grain-oriented silicon steel sheet is obtained by growing crystal grains of {110} <001> orientation, known as Goss orientation, by secondary recrystallization.

[0005] The following processes are involved in the production of a grain-oriented silicon steel sheet: heating and rolling at high temperature a silicon steel slab containing inhibitors required for secondary recrystallization, such as precipitates of MnS, MnSe, AlN and the like; cold-rolling the silicon steel sheet at low temperature at least once, or two or more times with intermediate annealing, to attain a final thickness; decarburization annealing the silicon steel sheet; applying an annealing separating agent such as MgO or the like to the steel sheet; and final annealing in the coil shape. Secondary recrystallization occurs during the final annealing process. An insulating coating comprising forsterite also forms during the final annealing process. Additional annealing after hot-rolling or during cold-rolling may be incorporated, and cold-rolling temperature may be raised as necessary.

[0006] Achieving further improvement in the magnetic properties requires a higher degree of secondary crystal grain growth in the Goss orientation. An effective means for producing such a result is to increase the rolling reduction to between 80-95% during final cold-rolling. However, when the rolling reduction during final cold-rolling reaches 80-95%, secondary recrystallization becomes very unstable, particularly in sheet steel less than 0.23 mm thick.

[0007] As a means for stabilizing secondary recrystallization when an increased rolling reduction is used during final cold-rolling, Japanese Patent Publication No. JP-B- 62-50529 (US-A-4 692 193) discloses a limited decarburization using AlN and MnS as principal inhibitors, such that carbon content is reduced by 0.0070 to 0.030 wt% after the hot-rolling process and before the cold-rolling process. However, B_8 of the resulting products is only 1.92T on average, thus the desired value of 1.92T cannot be consistently obtained. Furthermore, the prior art does not disclose materials utilizing AlN and MnSe as principal inhibitors.

[0008] Because the coexistence of AlN and MnSe enables multimodal precipitation, AlN and MnSe can finely disperse, thereby enhancing the inhibition effect. However, the presence of MnSe also renders insulating coating formation more difficult.

[0009] Japanese Patent Laid-Open No. JP-A-4-202713 (EP-A-488726) discloses that controlling ambient temperature within a suitable range during the temperature elevation and soaking temperature in the decarburization annealing process improves coating properties and magnetic properties. The effects of oxides on the steel surface before the temperature increase, however, is not considered. Further, when this prior art technique is applied to materials containing AlN and MnSe as principal inhibitors, magnetic properties over the entire product coil are inconsistent because secondary recrystallization at the middle portion of the coil is unstable.

[0010] As described above, no method for producing a coil-shaped, grain-oriented silicon steel sheet which possesses consistently excellent and stable magnetic properties has been found where AlN and MnSe are employed as inhibitors to promote high magnetic induction.

[0011] From EP-A-488726, there are known several decarburization schemes for electrical steel sheets. A ratio of steam partial pressure to hydrogen partial pressure of from 0.35 to 0.60 is disclosed for the step of temperature elevation during decarburization annealing. Said partial pressure ratio can be increased up to 0.5 during a subsequent soaking period during said decarburization annealing.

55 SUMMARY OF THE INVENTION

[0012] It is an object of this invention to provide a production method for a grain-oriented silicon steel sheet having

consistently excellent and stable magnetic properties in the coil shape and having high magnetic induction through the utilization of AlN and MnSe as principal inhibitors.

[0013] This invention is directed to the stabilization of magnetic properties at a high-quality level by stabilizing secondary recrystallization. The invention achieves stable secondary recrystallization by promoting the integration of secondary crystallized grain to the Goss orientation by raising the rolling reduction in the final cold-rolling to about 80-95%, decreasing oxide content before elevating the temperature for the decarburization annealing process, and controlling oxide composition and morphology formed at an early stage adjacent to the iron matrix-oxide interface by decreasing atmospheric oxidization which occurs during the temperature elevation phase in the decarburization annealing process.

[0014] According to the present invention, the above object is achieved by a method as defined in claim 1.

[0015] With specific regard to a grain oriented silicon steel sheet having a copper contents of from 0.03 to 0.20 wt%, the above object is achieved by the subject matter of claim 2.

[0016] The invention promotes the formation of stable secondary recrystallized grains in different coils or at different places in the same coil, thereby depressing undesirable fluctuations in magnetic properties.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017]

Fig. 1 is a graph illustrating the correlation between magnetic induction and the oxide content in the steel sheet before the temperature elevation phase of a decarburization annealing process;

Fig. 2 is a graph showing the correlation between the oxidation atmosphere and imperfect secondary recrystallization rate during the temperature elevation phase of a decarburization annealing process; and

Fig. 3 is a graph showing the correlation between the oxidation atmosphere and imperfect secondary recrystallization rate during the temperature elevation phase of a decarburization annealing process in case of Cu-added steel sheet.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0018] We have intensively studied secondary recrystallization behavior of the grain-oriented silicon steel sheet when the rolling reduction at the final cold-rolling is raised to about 80-95% while using MnSe and AlN inhibitors. We have discovered that surface oxides, which form near the iron matrix interface during the temperature elevation phase in a decarburization annealing process, affect dissociation and surface reaction of inhibitors during secondary recrystallization annealing and thus determine whether secondary recrystallization will occur. These effects are remarkably strong at the middle section of coil during the final annealing process carried out in the coil shape because of inadequate gas flow to those areas.

[0019] Accordingly, by controlling the oxide content on the steel surface before the temperature elevation phase of a decarburization annealing process, and by controlling the decarburization annealing atmosphere ("oxidizing atmosphere") during the temperature elevation phase of a decarburization annealing process, a coil having stable and consistent magnetic properties can be produced as a result of (1) the uniform surface oxide formation near the iron matrix interface, and (2) stable secondary recrystallization at the middle section of the coil.

[0020] The investigations through which the present invention was discovered will now be detailed.

[0021] First, the effect of oxide content formed on the steel sheet surface before the temperature elevation phase of a decarburization annealing process on the stability of the magnetic properties of the products and the secondary recrystallization were investigated. The oxide content formed on the steel sheet surface represents the oxygen content (g/m^2) per unit area existing in the area from the sheet surface to the $0.8 \mu\text{m}$ depth of the sheet. The oxides are formed as inner oxide layers during intermediate annealing and cold-rolling, which generally involve heat generation by the processing, and during rolling at high temperature and aging. The oxide content is usually about 0.1 to 0.2 g/m^2 immediately after the final cold-rolling.

[0022] The experimental procedure is as follows: A slab containing 0.078 wt% C, 3.25 wt% Si, 0.08 wt% Mn, 0.022 wt% Se, 0.024 wt% Al, and 0.0090 wt% N was rolled at high temperature (hot-rolled) to form a hot-rolled sheet; The hot-rolled sheet was rolled at low temperature (cold-rolled), annealed at 1100°C , and again cold-rolled at 85% of final rolling reduction to form a cold-rolled sheet 0.23 mm thick. After decarburization annealing and applying an annealing separation agent, the final annealing was performed to form a final product. The magnetic properties of the final product were then measured. The oxide content remaining on the surface of resulting steel sheet was controlled by various acid cleaning and brushing techniques. In the decarburization annealing process, the oxidizing atmosphere, i.e. the ratio of the steam partial pressure to the hydrogen partial pressure ($P(\text{H}_2\text{O})/P(\text{H}_2)$), during the temperature elevation between 500 and 750°C was controlled to 0.45. The soaking temperature was 840°C , during which $P(\text{H}_2\text{O})/P(\text{H}_2)$ was 0.55. The results are shown in Fig. 1.

[0023] Fig. 1 shows that by controlling the oxide content on the steel surface to about 0.02 to 0.10 g/m², the magnetic induction (B_8) exceeds 1.92T, thereby indicating stabilized secondary recrystallization.

[0024] Then, the effects of the atmosphere during the temperature elevation in a decarburization annealing process were investigated. Maintaining the atmosphere over a steel surface temperature range between about 500 and 750°C before reaching the decarburization temperature range is particularly important as is demonstrated in the following experiments.

[0025] A slab containing 0.078 wt% C, 3.25 wt% Si, 0.08 wt% Mn, 0.022 wt% Se, 0.024 wt% Al, and 0.0090 wt% N was hot-rolled to make a hot-rolled sheet. The hot-rolled sheet was cold-rolled, annealed at 1100°C, and again cold-rolled at 85% of final rolling reduction to make a cold-rolled sheet 0.23 mm thick.

[0026] The oxide content before decarburization annealing was adjusted to 0.05 g/m². During the decarburization annealing process, the oxidizing atmosphere $P(H_2O)/P(H_2)$ over the elevating temperature range of 500 to 750°C was controlled to various values. $P(H_2O)/P(H_2)$ in the temperature range from 750 to 850°C was controlled to 0.6.

[0027] After decarburization annealing and applying an annealing separation agent, a final annealing was performed on the cold-rolled sheet to produce a final product. The magnetic properties of the final product were then measured.

[0028] Imperfect secondary recrystallization was indicated by a magnetic induction (B_8) of less than 1.92T. In Fig. 2, the imperfect secondary recrystallization rate represents the ratio of the length of the imperfectly secondary recrystallized portion of the coil to the entire coil length. Fig. 2 clearly shows that the imperfect secondary recrystallization rate increases when $P(H_2O)/P(H_2)$ is outside the range of about 0.3 to 0.5 during the temperature elevation phase (between about 500 and 750°C) of the decarburization annealing. Thus, stable secondary recrystallization essentially requires controlling $P(H_2O)/P(H_2)$ during the temperature elevation phase of the decarburization annealing process in the range of about 0.3 to 0.5.

[0029] Stabilization of the secondary recrystallization by controlling the surface oxides before the decarburization annealing temperature elevation phase, and by controlling the oxidizing atmosphere during that elevation phase, is believed to occur through the following mechanism.

[0030] Oxides of Fe and Si having various compositions (e.g., silica and fayalite) are formed in various morphologies (e.g., epitaxial growth on the crystal axis of the matrix iron and dispersion in an amorphous state) on the steel sheet surface after decarburization annealing. In the subsequent annealing process, inhibitors in the steel sheet migrate or dissociate. The migration or dissociation is carried out through oxides on the steel sheet, depending on the atmosphere. Through migration or dissociation of the inhibitors, grain boundary migration becomes feasible so that secondary recrystallization occurs. Therefore, the secondary recrystallization greatly depends on the oxides on the steel sheet surface after decarburization annealing, and on the atmosphere.

[0031] Accordingly, stabilization of oxide composition and morphology on the steel sheet surface after decarburization annealing stabilizes secondary recrystallization. The factor controlling the oxide composition and morphology on the steel sheet surface after decarburization annealing is the state of oxides at the iron matrix-oxide interface of the steel sheet, i.e. initial oxides. Although it is yet unclear which compositions and morphologies of the initial oxides are preferred, suitable surface conditions can be obtained by controlling the oxide content before the temperature elevation phase of a decarburization annealing process and the oxidizing atmosphere during that temperature elevation phase, so that secondary recrystallization becomes stable. The effect is especially remarkable in the middle section of the coil where gas flow is low, particularly during final annealing.

[0032] The effect of adding Cu to the steel on the stabilization of secondary recrystallization will now be detailed.

[0033] We have studied various means for spreading the range of the oxidizing atmosphere during the temperature elevation phase of decarburization annealing, and found that steels containing about 0.03 to 0.20 wt% Cu permit secondary recrystallized grain to be stably obtained over a wider range of oxidizing atmosphere $P(H_2O)/P(H_2)$.

[0034] A slab containing 0.078 wt% C, 3.25 wt% Si, 0.08 wt% Mn, 0.022 wt% Se, 0.024 wt% Al, 0.0090 wt% N, and 0.12 wt% Cu was hot-rolled to make a hot-rolled sheet. The hot-rolled sheet was cold-rolled, annealed at 1100°C, and again cold-rolled at 85% of final rolling reduction to make a cold-rolled sheet 0.23 mm thick. After decarburization annealing and applying an annealing separation agent, a final annealing was performed to make a final product. The magnetic properties of the final product were then measured.

[0035] The oxide content before decarburization annealing was adjusted to 0.05 g/m². During the decarburization annealing process, $P(H_2O)/P(H_2)$ over the elevating temperature range of 500 to 750°C was controlled to various values. $P(H_2O)/P(H_2)$ in the temperature range from 750 to 850°C was maintained at 0.6. The results of the imperfect secondary recrystallization rate of various final products containing Cu are shown in Fig. 3.

[0036] Fig. 3 clearly shows that the preferable $P(H_2O)/P(H_2)$ range over the decarburization annealing temperature elevation phase range of 500 to 750°C is from about 0.2 to 0.65, which enables stable and consistently excellent magnetic properties to be obtained.

[0037] However, a Cu content over about 0.20 wt% causes Cu-Se to precipitate, which has a harmful effect on secondary recrystallization and deteriorates magnetic properties. These effects are not seen when less than about 0.03 wt% is added. Such results suggest that Cu affects surface oxide formation.

[0038] The quantity limits on elemental components of the present invention will now be explained.

[0039] C content in the silicon steel slab should be in a range of 0.04 to 0.12 wt%. Steels with C content under about 0.04 wt% do not form suitable textures during the hot-rolling process; consequently, the final product does not possess suitable magnetic properties. On the other hand, steels with C content over about 0.12 wt% are hard to satisfactorily decarburize during the decarburization annealing process; therefore, secondary recrystallization cannot be normally carried out.

[0040] The Si content in the steel slab should be in a range of 2.0 to 4.5 wt%. A final product containing less than about 2.0 wt% Si does not possess satisfactory magnetic properties. On the other hand, when Si content is over about 4.5 wt%, industrial working is difficult because of poor secondary recrystallization and poor formability.

[0041] The silicon steel slab containing the above components should also contain the components described below.

[0042] The steel should contain 0.02 to 0.15 wt% Mn. An Mn content under about 0.02 wt% causes poor formability during hot-rolling and markedly poor surface characteristics. Further, the lack of MnSe inhibitor essential for secondary recrystallization causes imperfect secondary recrystallization. On the other hand, when the Mn content exceeds about 0.15 wt%, the slab heating temperature during the hot-rolling process needs to be set at a higher temperature in order to completely form the solid solution of MnSe, thereby increasing processing costs while deteriorating the surface characteristics of the slab.

[0043] The Se content in the steel should be in a range of 0.005 to 0.06 wt%. An Se content less than about 0.005 wt% causes imperfect secondary recrystallization due to the lack of MnSe inhibitor. On the other hand, when the Se content exceeds about 0.06 wt%, the slab heating temperature during the hot-rolling process needs to be raised in order to completely form the solid solution of MnSe, thereby increasing processing costs while deteriorating the surface characteristics of the slab.

[0044] The Al content of the slab should be in a range of 0.010 to 0.06 wt%. An Al content less than about 0.010 wt% causes imperfect secondary recrystallization due to the lack of AlN inhibitor. On the other hand, when Al content exceeds about 0.06 wt%, the growth of AlN grain after hot-rolling decreases the action of the inhibitor such that normal secondary recrystallization will not occur.

[0045] The N content in the steel should be in a range of 0.0030 to 0.0120 wt%. An N content less than about 0.0030 wt% causes imperfect secondary recrystallization due to the lack of AlN inhibitor. On the other hand, when N content exceeds about 0.0120 wt%, surface blisters formed during the slab heating process deteriorate the surface characteristics.

[0046] As described above, the grain-oriented silicon steel material may preferably contain 0.03 to 0.20 wt% Cu. The addition of Cu enables secondary recrystallization to be carried out over a wider oxidation atmosphere range in terms of $P(H_2O)/P(H_2)$, and promotes stable and excellent magnetic properties. However, a Cu content over about 0.20 wt% has a harmful influence on secondary recrystallization, thus leading to a lower B_8 value. The addition of less than about 0.03 wt% produces no significant effect.

[0047] The silicon steel slab having the above composition can be rolled at high temperature using conventional methods. After hot-rolling, cold-rolling is performed at least once, or twice or more with intermediate annealing between the cold-rollings, so that a desired sheet thickness is obtained. The rolling reduction during the final cold-rolling should range from about 80-95%. When the rolling reduction is less than about 80%, a highly-oriented sheet is not obtainable, while a rolling reduction over about 95% fails to cause secondary recrystallization.

[0048] The steel sheet rolled to the final product thickness must contain 0.02 to 0.10 g/m² of oxides on the surface before the decarburization annealing process. An oxide content outside of that range causes unstable initial oxidation and poor magnetic properties. The oxide content can be adjusted by controlling heating during the cold-rolling process, or by brushing or cleaning with acid during the final cold-rolling process.

[0049] In the decarburization annealing process, the steel temperature must be maintained in a range of 800 to 850°C for effective decarburization. A temperature below about 800°C causes a disadvantageously lowered decarburization rate as well as poor magnetic properties, while a temperature over about 850°C causes deterioration in coating properties and in imperfect secondary recrystallization.

[0050] The decarburization annealing oxidizing atmosphere during the steel temperature elevation phase from about 500 to 750°C (before reaching the decarburization annealing temperature range) is important, so $P(H_2O)/P(H_2)$ must be controlled within a range of 0.3 to 0.5, or 0.2 to 0.65 in the case the steel has a Cu content in accordance with the present invention. A $P(H_2O)/P(H_2)$ less than about 0.3 or 0.2 tends to cause imperfect secondary recrystallization. On the other hand, when $P(H_2O)/P(H_2)$ is over about 0.5 or 0.65, secondary recrystallization becomes imperfect, and defects form on the steel sheet because of sticking and piling of the oxides, which formed in the furnace due to the excessive oxidizing atmosphere.

[0051] In the steel temperature range of 750 to 850°C during decarburization annealing, $P(H_2O)/P(H_2)$ must be controlled within a range of 0.5 to 0.8 for effective decarburization and satisfactory coating. Deviation from that $P(H_2O)/P(H_2)$ range causes poor magnetic properties and poor coating appearance.

[0052] The present invention is also effective in magnetic domain refined steel sheets.

[0053] The invention will now be described through illustrative examples. The examples are not intended to limit the scope of the invention defined in the appended claims.

EXAMPLE 1

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[0054] Hot-rolled sheets were made from a steel slab containing 0.078 wt% C, 3.25 wt% Si, 0.08 wt% Mn, 0.022 wt% Se, 0.024 wt% Al, and 0.0090 wt% N by hot-rolling. The sheets were cold-rolled, annealed at 1,100°C (intermediate annealing), and again cold-rolled at 85% of the final rolling reduction to obtain a steel sheet 0.23 mm thick. Then, the surface oxide contents of the steels were varied as shown in Table 1 by cleaning and brushing. The following decarburization annealing process was carried out by choosing among four oxidizing atmosphere levels, i.e. $P(H_2O)/P(H_2) = 0.2, 0.4, 0.5$ and 0.6 , respectively, by controlling steam content in the oxidizing atmosphere during the temperature elevation phase from 500 to 750°C. Then, a soaking process was carried out at 835°C where $P(H_2O)/P(H_2) = 0.5, 0.6$ or 0.7 . The resulting steel sheets were evaluated in regard to secondary recrystallization and magnetic properties. The results are shown in Table 1.

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[0055] In the evaluation, core loss values were continuously measured in the longitudinal direction of the coil. Where the core loss value reached a threshold level defined for each sheet thickness, the secondary recrystallization was deemed to be perfect. The excellent article rate refers to the ratio of the longitudinal length of the coil which attains the defined threshold level to the total coil length. "Normal portion" refers to the portion of the coil which has attained the defined threshold level.

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Table 1

Oxide content before the temperature elevation phase of decarburization annealing (g/m^2)	Atmosphere during the temperature elevation phase of decarburization annealing $P(\text{H}_2\text{O})/P(\text{H}_2)$	Atmosphere during soaking $P(\text{H}_2\text{O})/P(\text{H}_2)$	Secondary recrystallization	Excellent rate (%)	Magnetic induction of normal portion $B_8(\text{T})$	Remarks
0.005	0.2	0.6	imperfect	60	1.92	unsatisfactory
0.005	0.4	0.6	imperfect	65	1.88	unsatisfactory
0.005	0.6	0.6	imperfect	50	1.89	unsatisfactory
0.05	0.2	0.6	imperfect	70	1.93	unsatisfactory
0.05	0.4	0.5	perfect	100	1.93	good
0.07	0.4	0.6	perfect	100	1.94	good
0.05	0.6	0.5	imperfect	80	1.90	unsatisfactory
0.4	0.4	0.6	perfect	50	1.87	unsatisfactory
0.02	0.5	0.7	perfect	100	1.94	good
0.013	0.5	0.7	perfect	100	1.93	good

EXAMPLE 2

5 **[0056]** Hot-rolled sheets were made from a steel slab containing 0.079 wt% C, 3.25 wt% Si, 0.08 wt% Mn, 0.023 wt% Se, 0.025 wt% Al, 0.0085 wt% N, and 0.16 wt% Cu by hot-rolling. The sheets were cold-rolled, annealed at 1,100°C (intermediate annealing), and again cold-rolled at 85% of final rolling reduction to obtain a steel sheet 0.23 mm thick. Then, the surface oxide content of thus produced steel sheet was adjusted to 0.05 g/m² by cleaning and brushing. The following decarburization annealing process was carried out by choosing among three oxidizing atmosphere levels, i. e. $P(H_2O)/P(H_2) = 0.2, 0.4$ and 0.6 , respectively, by controlling steam content in the oxidizing atmosphere during the temperature elevation phase from 500 to 750°C. Then, a soaking process was carried out at 835°C under the condition of $P(H_2O)/P(H_2) = 0.5$ or 0.6 . Evaluations of the secondary recrystallization state, excellent article rate, and the magnetic properties at the normal portion were undertaken, and the results are shown in Table 2.

10 **[0057]** Table 2 indicates that excellent magnetic properties are stably obtainable when the steel contains Cu in accordance with the present invention even when $P(H_2O)/P(H_2) = 0.2$ or 0.6 during the steel temperature elevation phase from 500 to 750°C of the decarburization annealing.

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Table 2

Oxide content before the temperature elevation phase of decarburization annealing (g/m^2)	Atmosphere during the temperature elevation phase of decarburization annealing $P(\text{H}_2\text{O})/P(\text{H}_2)$	Atmosphere during soaking $P(\text{H}_2\text{O})/P(\text{H}_2)$	Secondary recrystallization	Excellent rate (%)	Magnetic induction of normal portion $B_8(\text{T})$	Remarks
0.05	0.2	0.6	perfect	100	1.93	good
0.05	0.4	0.5	perfect	100	1.94	good
0.05	0.4	0.6	perfect	100	1.94	good
0.05	0.6	0.5	perfect	100	1.93	good

Claims

1. A method for producing a grain-oriented silicon steel sheet which includes:

5 producing a silicon steel slab having a silicon steel slab composition comprising 0.04 wt% to 0.12 wt% carbon and 2.0 wt% to 4.5 wt% silicon said silicon steel slab composition comprises, as inhibitor forming components, 0.02 to 0.15 wt% Mn, 0.005 to 0.060 wt% Se, 0.010 to 0.06 wt% Al, and 0.0030 to 0.0120 wt% N, the balance apart from inevitable impurities being Fe;
 hot-rolling said silicon steel slab to produce a silicon steel sheet;
 10 cold-rolling said silicon steel sheet at least once, including a final 80 - 95% cold-rolling, to produce a silicon steel sheet; decarburization annealing said silicon steel sheet, said decarburization annealing including a temperature elevation phase and an oxidizing atmosphere, to produce decarburized silicon steel sheet;
 applying an annealing separating agent to said decarburized silicon steel sheet; and
 15 final annealing said decarburized silicon steel sheet to produce said grain-oriented silicon steel sheet; the steps which comprise:

controlling the oxide content on said silicon steel sheet surface within 0.02 to 0.10 g/m² in a depth up to 0.8 μm before said temperature elevation phase in said decarburization annealing; maintaining the ratio of steam partial pressure to hydrogen partial pressure in said oxidizing atmosphere within a range of 0.3 to 0.5 when said silicon steel sheet has a surface **[deletion(s)]** temperature ranging from 500 to 750°C during said temperature elevation phase in said decarburization annealing and by decreasing the atmospheric oxidation occurring in said temperature elevation phase in respect to the subsequent step of said decarburization; and maintaining the ratio of steam partial pressure to hydrogen partial pressure in said oxidizing atmosphere within a range of 0.5. to 0.8 when said silicon steel sheet has a surface temperature ranging from 750 to 850°C during said decarburization annealing.

2. A method for producing a grain-oriented silicon steel sheet which includes:

30 producing a silicon steel slab having a silicon steel slab composition comprising 0.04 wt% to 0.12 wt% carbon and 2.0 wt% to 4.5 wt% silicon, said silicon steel slab composition comprises 0.03 to 0.20 wt% Cu, and, as inhibitor forming components, 0.02 to 0.15 wt% Mn, 0.005 to 0.060 wt% Se, 0.010 to 0.06 wt% Al, and 0.0030 to 0.0120 wt% N, the balance apart from inevitable impurities being Fe;
 hot-rolling said silicon steel slab to produce a silicon steel sheet;
 35 cold-rolling said silicon steel sheet at least once, including a final 80 - 95% cold-rolling to produce a silicon steel sheet;
 decarburization annealing said silicon steel sheet, said decarburization annealing including a temperature elevation phase and an oxidizing atmosphere to produce a decarburized silicon steel sheet;
 40 applying an annealing separating agent to said decarburized silicon steel sheet; and
 final annealing said decarburized silicon steel sheet to produce said grain-oriented silicon steel sheet; the steps which comprise:

controlling the oxide content on said silicon steel sheet surface within 0.02 to 0.10 g/m² in a depth up to 0.8 μm before said temperature elevation phase in said decarburization annealing; maintaining the ratio of steam partial pressure to hydrogen partial pressure in said oxidizing atmosphere within a range of 0.2 to 0.65 when said silicon steel sheet has a surface temperature ranging from 500 to 750°C during said temperature elevation phase in said decarburization annealing and by keeping decreased the atmospheric oxidation occurring in said temperature elevation phase in respect to the subsequent step of said decarburization; and maintaining the ratio of steam partial pressure to hydrogen partial pressure in said oxidizing atmosphere within a range of 0.5 to 0.8 when said silicon steel sheet has a surface temperature ranging from 750 to 850° during said decarburization annealing.

3. The method according to claim 1, further comprising:

55 controlling said silicon steel slab composition to comprise 0.04 to 0.12 wt% C and 2.0 to 4.5 wt% Si;
 performing said final cold-rolling with a rolling reduction ranging from 80 - 95%; and
 conducting said decarburization annealing at a temperature between 800 to 850°C.

4. The method according to claim 2, further comprising:

controlling said silicon steel slab composition to comprise 0.04 to 0.12 wt% C and 2.0 to 4.5 wt% Si;
performing said final cold-rolling with a rolling reduction ranging from 80 - 95%; and
conducting said decarburization annealing at a temperature between 800 to 850°C.

Patentansprüche

1. Verfahren zum Herstellen eines korn-orientierten Siliziumstahlblechs, das umfasst:

Herstellen einer Siliziumstahlbramme, die eine Siliziumstahlbrammenzusammensetzung besitzt, aufweisend 0,04 Gew.-% bis 0,12 Gew.-% an Kohlenstoff und 2,0 Gew.-% bis 4,5 Gew.-% an Silizium, wobei die Siliziumstahlbrammenzusammensetzung, als einen Inhibitor bildende Komponenten, aufweist 0,02 bis 0,15 Gew.-% an Mn, 0,005 bis 0,060 Gew.-% an Se, 0,010 bis 0,06 Gew.-% an Al und 0,0030 bis 0,0120 Gew.-% an N, wobei der Rest, außer unvermeidbaren Verunreinigungen, Fe ist;
Warmwalzen der Siliziumstahlbramme, um ein Siliziumstahlblech herzustellen; Kaltwalzen des Siliziumstahlblechs mindestens einmal, umfassend ein 80-95% Endkaltwalzen, um ein Siliziumstahlblech herzustellen;
Dekarbonisierungsglühen des Siliziumstahlblechs, wobei das Dekarbonisierungsglühen eine Temperaturanhebungsphase und eine oxidierende Atmosphäre umfasst, um dekarbonisiertes Siliziumstahlblech herzustellen;
Aufbringen eines Glühseparationsmittels auf das dekarbonisierte Siliziumstahlblech; und
Endglühen des dekarbonisierten Siliziumstahlblechs, um das korn-orientierte Siliziumstahlblech herzustellen; wobei die Schritte aufweisen:

Kontrollieren des Oxidgehalts auf der Siliziumstahlblechoberfläche innerhalb von 0,02 bis 0,10 g/m² in einer Tiefe bis zu 0,8 µm vor der Temperaturanhebungsphase bei dem Dekarbonisierungsglühen;
Beibehalten des Verhältnisses eines Dampfpartialdrucks zu einem Wasserstoffpartialdruck in der oxidierenden Atmosphäre innerhalb eines Bereichs von 0,3 bis 0,5, wenn das Siliziumstahlblech eine Oberflächentemperatur besitzt, die von 500 bis 750°C reicht, während der Temperaturanhebungsphase bei dem Dekarbonisierungsglühen und durch Verringern der atmosphärischen Oxidation, die in der Temperaturanhebungsphase auftritt, in Bezug auf den darauffolgenden Schritt der Dekarbonisierung; und Beibehalten des Verhältnisses des Dampfpartialdrucks zu dem Wasserstoffpartialdruck in der oxidierenden Atmosphäre innerhalb eines Bereichs von 0,5 bis 0,8, wenn das Siliziumstahlblech eine Oberflächentemperatur besitzt, die von 750 bis 850°C reicht, während des Dekarbonisierungsglühens.

2. Verfahren zum Herstellen eines korn-orientierten Siliziumstahlblechs, das umfasst:

Herstellen einer Siliziumstahlbramme, die eine Siliziumstahlbrammenzusammensetzung besitzt, aufweisend 0,04 Gew.-% bis 0,12 Gew.-% an Kohlenstoff und 2,0 Gew.-% bis 4,5 Gew.-% an Silizium, wobei die Siliziumstahlbrammenzusammensetzung 0,03 bis 0,20 Gew.-% an Cu und, als einen Inhibitor bildende Komponenten, 0,02 bis 0,15 Gew.-% an Mn, 0,005 bis 0,060 Gew.-% an Se, 0,010 bis 0,06 Gew.-% an Al und 0,0030 bis 0,0120 Gew.-% an N, wobei der Rest, neben unvermeidbaren Verunreinigungen, Fe ist;
Warmwalzen der Siliziumstahlbramme, um ein Siliziumstahlblech herzustellen; Kaltwalzen des Siliziumstahlblechs mindestens einmal, umfassend ein 80-95% Endkaltwalzen, um ein Siliziumstahlblech herzustellen;
Dekarbonisierungsglühen des Siliziumstahlblechs, wobei das Dekarbonisierungsglühen eine Temperaturanhebungsphase und eine oxidierende Atmosphäre umfasst, um ein dekarbonisiertes Siliziumstahlblech herzustellen;
Aufbringen eines Glühseparationsmittels auf das dekarbonisierte Siliziumstahlblech; und
Endglühen des dekarbonisierten Siliziumstahlblechs, um das korn-orientierte Siliziumstahlblech herzustellen; wobei die Schritte aufweisen:

Kontrollieren des Oxidgehalts auf der Siliziumstahlblechoberfläche innerhalb von 0,02 bis 0,10 g/m² in einer Tiefe bis zu 0,8 µm vor der Temperaturanhebungsphase bei dem Dekarbonisierungsglühen;
Beibehalten des Verhältnisses des Dampfpartialdrucks zu dem Wasserstoffpartialdruck in der oxidierenden Atmosphäre innerhalb eines Bereichs von 0,2 bis 0,65, wenn das Siliziumstahlblech eine Oberflächentemperatur besitzt, die von 500 bis 750°C reicht, während der Temperaturanhebungsphase bei dem Dekarbonisierungsglühen und durch verringert Halten, der atmosphärischen Oxidation, die in der Tem-

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peraturanhebungsphase auftritt, in Bezug auf den darauffolgenden Schritt der Dekarbonisierung; und Beibehalten des Verhältnisses des Dampfpartialdrucks zu dem Wasserstoffpartialdruck in der oxidierenden Atmosphäre innerhalb eines Bereichs von 0,5 bis 0,8, wenn das Siliziumstahlblech eine Oberflächentemperatur besitzt, die von 750 bis 850°C reicht, während des Dekarbonisierungsglühens.

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3. Verfahren nach Anspruch 1, das weiterhin aufweist:

Kontrollieren der Siliziumstahlbrammenzusammensetzung, um 0,04 bis 0,12 Gew.-% an C und 2,0 bis 4,5 Gew.-% an Si aufzuweisen;

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Durchführen des Endkaltwalzens mit einer Walzreduktion, die von 80 - 95% reicht; und
Durchführen des Dekarbonisierungsglühens bei einer Temperatur zwischen 800 und 850°C.

4. Verfahren nach Anspruch 2, das weiterhin aufweist:

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Kontrollieren der Siliziumstahlbrammenzusammensetzung, um 0,04 bis 0,12 Gew.-% an C und 2,0 bis 4,5 Gew.-% an Si aufzuweisen;

Durchführen des Endkaltwalzens mit einer Walzreduktion, die von 80-95% reicht; und
Durchführen des Dekarbonisierungsglühens bei einer Temperatur zwischen 800 und 850°C.

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Revendications

1. Procédé de fabrication d'une tôle d'acier au silicium à grains orientés incluant :

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la fabrication d'une brame d'acier au silicium ayant une composition de brame d'acier au silicium comprenant entre 0,04 % en poids et 0,12 % en poids de carbone et entre 2,0 % en poids et 4,5 % en poids de silicium,

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ladite composition de brame d'acier au silicium comprenant en tant que composants formant inhibiteurs entre 0,02 % en poids et 0,15 % en poids de Mn, entre 0,005 % en poids et 0,060 % en poids de Se, entre 0,010 % en poids et 0,06 % en poids de Al et entre 0,0030 % en poids et 0,0120 % en poids de N, le reste étant Fe, à part les impuretés inévitables ;

le laminage à chaud de ladite brame d'acier au silicium pour fabriquer une tôle d'acier au silicium ;

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le laminage à froid de ladite tôle d'acier au silicium, au moins une fois, incluant un laminage à froid final entre 80 et 95 %, pour fabriquer une tôle d'acier au silicium ;

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un recuit de décarburation de ladite tôle d'acier au silicium, ledit recuit de décarburation incluant une phase d'élévation de la température et une atmosphère oxydante, pour fabriquer une tôle d'acier au silicium décarburée ;

l'application d'un agent de séparation de recuit sur ladite tôle d'acier au silicium décarburée ; et

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un recuit final de ladite tôle d'acier au silicium décarburée pour fabriquer ladite tôle d'acier au silicium à grains orientés ;

les étapes comprenant :

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la régulation de la teneur en oxydes sur la surface de ladite tôle d'acier au silicium entre 0,02 et 0,10 g/m², sur une épaisseur pouvant atteindre 0,8 µm avant ladite phase d'élévation de la température pendant ledit recuit de décarburation ;

55

le maintien du rapport de la pression partielle de vapeur sur la pression partielle d'hydrogène dans ladite atmosphère oxydante dans une fourchette de 0,3 à 0,5 lorsque ladite tôle d'acier au silicium présente une température de surface comprise entre 500 et 750 °C au cours de ladite phase d'élévation de la température pendant ledit recuit de décarburation et en réduisant l'oxydation atmosphérique qui se produit au cours de la phase d'élévation de la température par rapport à l'étape suivante de ladite décarburation ;

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et le maintien du rapport de la pression partielle de vapeur sur la pression partielle d'hydrogène dans ladite atmosphère oxydante dans une fourchette de 0,5 à 0,8 lorsque ladite tôle d'acier au silicium présente une température de surface comprise entre 750 et 850 °C au cours dudit recuit de décarburation.

5 2. Procédé de fabrication d'une tôle d'acier au silicium à grains orientés incluant :

la fabrication d'une brame d'acier au silicium ayant une composition de brame d'acier au silicium comprenant entre 0,04 % en poids et 0,12 % en poids de carbone et entre 2,0 % en poids et 4,5 % en poids de silicium,

10 ladite composition de brame d'acier au silicium comprenant, entre 0,03 % en poids et 0,20 % en poids de Cu, et, en tant que composants formant inhibiteurs entre 0,02 % en poids et 0,15 % en poids de Mn, entre 0,005 % en poids et 0,060 % en poids de Se, entre 0,010 % en poids et 0,06 % en poids de Al et entre 0,0030 % en poids et 0,0120 % en poids de N, le reste étant Fe, à part les impuretés inévitables ;

15 le laminage à chaud de ladite brame d'acier au silicium pour fabriquer une tôle d'acier au silicium ;

le laminage à froid de ladite tôle d'acier au silicium, au moins une fois, incluant un laminage à froid final entre 80 et 95 %, pour fabriquer une tôle d'acier au silicium ;

20 un recuit de décarburation de ladite tôle d'acier au silicium, ledit recuit de décarburation incluant une phase d'élévation de la température et une atmosphère oxydante, pour fabriquer une tôle d'acier au silicium décarburée ;

25 l'application d'un agent de séparation de recuit sur ladite tôle d'acier au silicium décarburée ; et

un recuit final de ladite tôle d'acier au silicium décarburée pour fabriquer ladite tôle d'acier au silicium à grains orientés ;

les étapes comprenant :

30 la régulation de la teneur en oxydes sur la surface de ladite tôle d'acier au silicium entre 0,02 et 0,10 g/m², sur une épaisseur pouvant atteindre 0,8 µm avant ladite phase d'élévation de la température pendant ledit recuit de décarburation ;

35 le maintien du rapport de la pression partielle de vapeur sur la pression partielle d'hydrogène dans ladite atmosphère oxydante dans une fourchette de 0,2 à 0,65 lorsque ladite tôle d'acier au silicium présente une température de surface comprise entre 500 et 750 °C au cours de ladite phase d'élévation de la température pendant ledit recuit de décarburation et en maintenant réduite l'oxydation atmosphérique qui se produit au cours de la phase d'élévation de la température par rapport à l'étape suivante de ladite décarburation ;

40 et maintien du rapport de la pression partielle de vapeur sur la pression partielle d'hydrogène dans ladite atmosphère oxydante dans une fourchette de 0,5 à 0,8 lorsque ladite tôle d'acier au silicium présente une température de surface comprise entre 750 et 850 °C au cours dudit recuit de décarburation.

45 3. Procédé selon la revendication 1, comprenant en outre :

la régulation de la composition de ladite brame d'acier au silicium pour qu'elle comprenne entre 0,04 % en poids et 0,12 % en poids de C et entre 2,0 % en poids et 4,5 % en poids de Si ;

50 la réalisation d'un laminage à froid final avec une réduction de laminage comprise entre 80 % et 95 % ; et

la conduite dudit recuit de décarburation à une température comprise entre 800 et 850 °C.

55 4. Procédé selon la revendication 2, comprenant en outre :

la régulation de la composition de ladite brame d'acier au silicium pour qu'elle comprenne entre 0,04 % en poids et 0,12 % en poids de C et entre 2,0 % en poids et 4,5 % en poids de Si ;

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la réalisation d'un laminage à froid final avec une réduction de laminage comprise entre 80 % et 95 % ; et

la conduite dudit recuit de décarburation à une température comprise entre 800 et 850 °C.

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FIG. 1

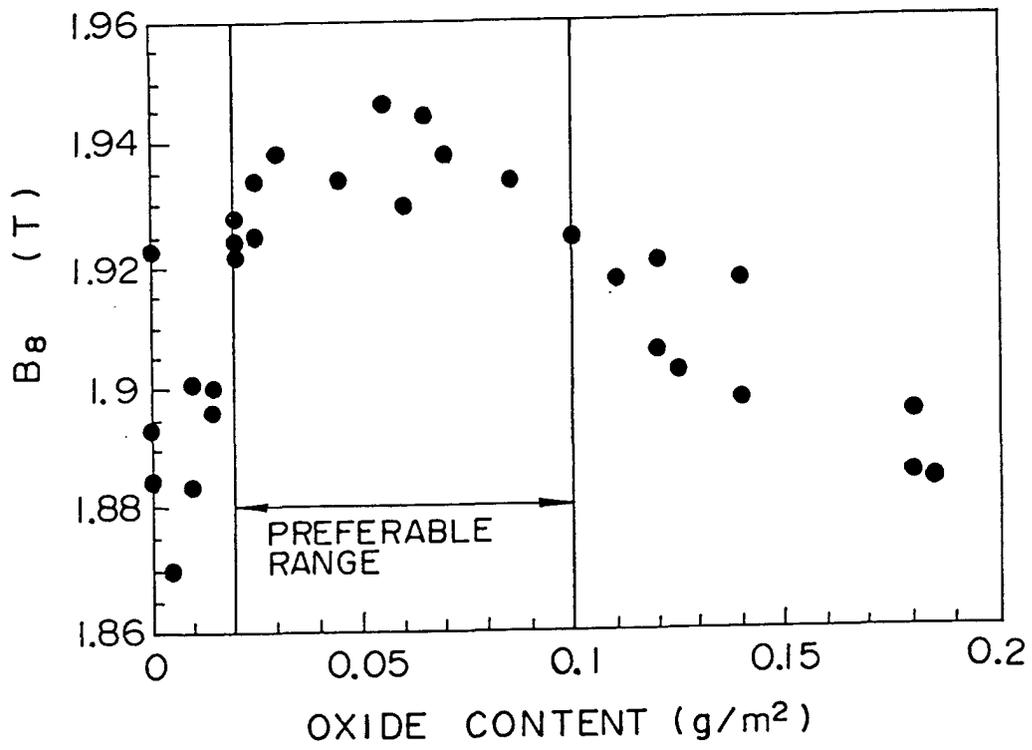


FIG. 2

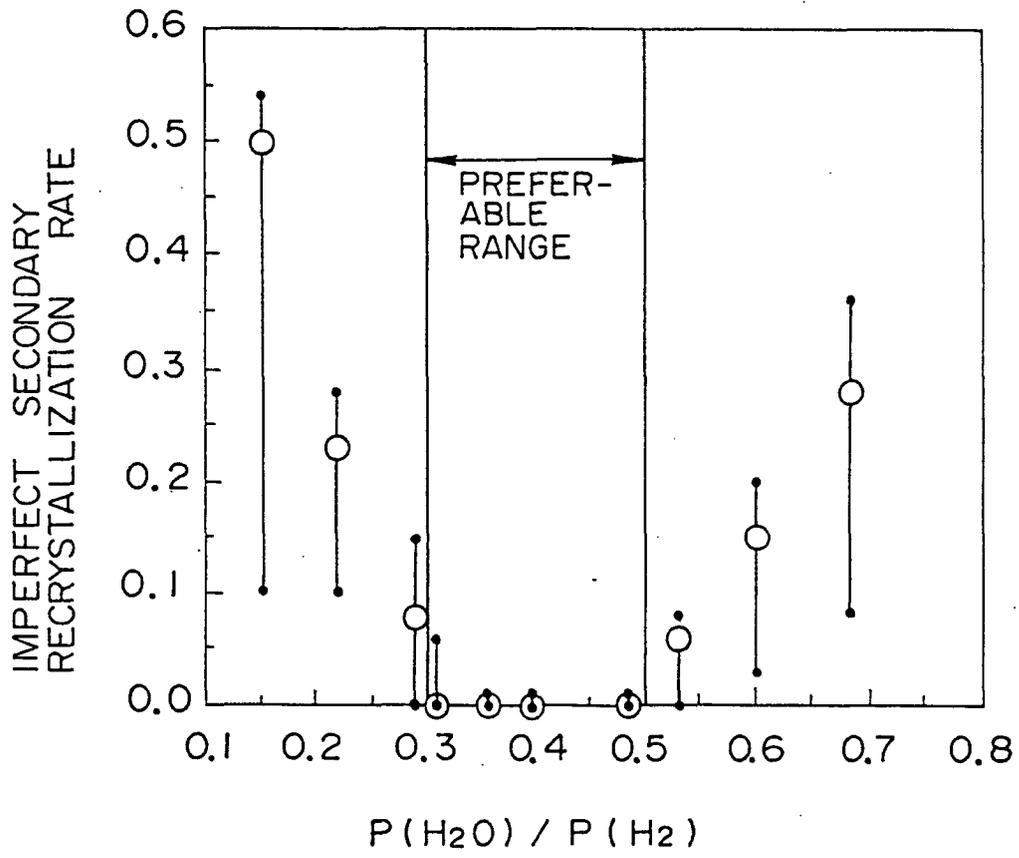


FIG. 3

