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(54) **HOT ROLLED ELECTRICAL STEEL SHEET EXCELLENT IN MAGNETIC CHARACTERISTICS
AND CORROSION RESISTANCE AND METHOD FOR PRODUCTION THEREOF**

HEISS GEWALZTES ELEKTROBLECH MIT HERVORRAGENDEN MAGNETISCHEN- UND
KORROSIONSEIGENSCHAFTEN UND VERFAHREN ZU DESSEN HERSTELLUNG

FEUILLE D'ACIER MAGNETIQUE LAMINEE A CHAUD PRESENTANT DES CARACTERISTIQUES
MAGNETIQUES ET UNE RESISTANCE A LA CORROSION EXCELLENTE, ET PROCEDE DE
FABRICATION CORRESPONDANT

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JP-A- 5 117 817 **JP-A- 5 306 437**
JP-A- 8 027 516 **JP-A- 62 077 420**

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- **PATENT ABSTRACTS OF JAPAN vol. 014, no. 246 (C-0722), 25 May 1990 (1990-05-25) & JP 02 066118 A (NKK CORP), 6 March 1990 (1990-03-06)**

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Description

TECHNICAL FIELD

5 **[0001]** This invention relates to a hot rolled electromagnetic steel sheet, and more particularly to a pure iron based hot rolled electromagnetic steel sheet having excellent magnetic properties, by aligning the <100> axis in a direction perpendicular to a sheet surface at as-rolled state in a high density and an excellent corrosion resistance. A method of producing such a steel sheet is also provided.

10 BACKGROUND ART

[0002] Silicon steel sheets having excellent electromagnetic properties have been used in a core for a transformer or a generator for some time. As such a silicon steel sheet there are two kinds, namely a unidirectional silicon steel sheet utilizing a secondary recrystallization to develop {110}<001> oriented grains or so-called Goss oriented grains, and a non-directional silicon steel sheet developing crystal grains with {100} face parallel to a sheet surface. Among these the non-directional silicon steel sheets have particularly good properties when a magnetic field is applied to various directions in the sheet surface and are frequently used in generators, electric motors and the like.

[0003] In order to produce the non-directional silicon steel sheet used for such applications, it has hitherto been required to conduct decarburization annealing in a controlled atmosphere, cross rolling for changing a rolling direction during the cold rolling or the like for gathering {100} face parallel to the sheet surface in a higher density.

[0004] For example, JP-A-1-108345 relating to silicon steel containing Si: 0.2-6.5 wt% and JP-A-4-224624 relating to steel containing Al+Si: 0.2-6.5 wt% disclose a technique wherein the steel is cold-rolled and annealed in a weak decarburizing atmosphere, for example, under vacuum of not more than 0.1 torr or in an atmosphere having a dew point of not more than 0°C and composed of one or more of H₂, He, Ne, Ar, Xe, Rn and N₂ to form α-single phase region in a zone corresponding to a depth of 5-50 μm from the sheet surface. The sheet is then annealed in a strong decarburizing atmosphere, for example, H₂ having a dew point of not less than -20°C or a gas obtained by adding an inert gas or CO, CO₂ to H₂ having a dew point of not less than -20°C at 650-900°C for 5-20 minutes to grow the α-single phase region formed on the surface layer portion into the inside in the thickness direction to thereby improve the magnetic properties.

[0005] Thus, complicated steps including the decarburization annealing are required in addition to the hot rolling - cold rolling steps for gathering the {100} face parallel to the sheet surface in a high density. EP-A-609 190 discloses a hot rolled steel strip with good electromagnetic properties in which the slab is rough rolled in the austenitic region, cooled into the ferritic region for finish rolling under lubricated rolls. Also, the conventional electromagnetic steel sheets including 3% Si steel are low in corrosion resistance, so that an insulating film having an excellent corrosion resistance is applied onto a final product, which is a factor raising the product cost.

[0006] However, it has recently been demanded to have high performance at a cheaper cost with the popularization of electrical goods, which is impossible - with the aforementioned conventional technique. Although it is considered to - simplify the production steps for satisfying the above demand, the conventional technique is difficult to enhance the gathering of {100} orientation parallel to the sheet surface as hot-rolled.

[0007] It is, therefore, desirable to provide a hot rolled electromagnetic steel sheet having improved magnetic properties and corrosion resistance by gathering the {100} orientation parallel to the sheet surface at a time of completing hot rolling, and a method of producing the same.

DISCLOSURE OF INVENTION

45 **[0008]** The inventors have made various studies for solving the above problems in the hot rolled electromagnetic steel sheet, and have found that the formation of {100} orientation parallel to sheet surface, i.e. <100>//ND orientation of the steel sheet (direction perpendicular to sheet surface) is promoted by highly purifying steel to form a pure iron based component composition and rationalizing hot rolling conditions (particularly rolling reduction at given temperature region, friction coefficient) and cooling rate at α-zone after hot rolling, and as a result the invention has been accomplished.

[0009] That is, the invention is a hot rolled electromagnetic steel sheet consisting of a super-high purity iron comprising Fe: not less than 99.95 mass%, C+N+S: not more than 10 mass ppm, O: not more than 50 mass ppm and the remainder being inevitable impurity, and having excellent magnetic properties and corrosion resistance, wherein the X-ray diffraction ratio I_{100}/I_0 of the steel sheet is not less than 21.

55 **[0010]** As a method of producing the above hot rolled electromagnetic steel sheet, the invention also proposes a method of producing a hot rolled electromagnetic steel sheet having excellent magnetic properties and corrosion resistance, characterized in that the X-ray diffraction ratio I_{100}/I_0 of the steel sheet is not less than 21 and the steel sheet

comprises a super-high purity iron comprising Fe: not less than 99.95 mass%, C+N+S: not more than 10 mass ppm, O: not more than 50 mass ppm and the remainder being inevitable impurity and is heated to γ -zone and subjected in this γ -zone to hot rolling at a total rolling reduction of not less than 50% and under condition that at least one pass has a friction coefficient between roll and rolling material of not more than 0.3 and thereafter cooled at an average cooling rate of 0.5-150°C per minute within a temperature range of A_{r3} transformation point $\sim 300^\circ\text{C}$.

[0011] As a preferable method, the invention proposes a method of producing a hot rolled electromagnetic steel sheet having excellent magnetic properties and corrosion resistance, characterized in that the X-ray diffraction ratio I_{100}/I_0 of the steel sheet is not less than 21 and the steel sheet comprises a super-high purity iron comprising Fe: not less than 99.95 mass%, C+N+S: not more than 10 mass ppm, O: not more than 50 mass ppm and the remainder being inevitable impurity and is heated to γ -zone and subjected in this γ -zone to hot rolling at a total rolling reduction of not less than 50% and under condition that at least one pass has a friction coefficient between roll and rolling material of not more than 0.3 and a strain rate of not less than 150 1/second and thereafter cooled at an average cooling rate of 0.5-150°C per minute within a temperature range of A_{r3} transformation point - 300°C .

BEST MODE FOR CARRYING OUT THE INVENTION

[0012] An embodiment of the invention will be described below.

[0013] Firstly, the reason for the limitation of the chemical composition in the pure iron based electromagnetic steel sheet according to the invention is described.

.Fe: not less than 99.95 mass%.

[0014] A raw material of high purity Fe is hot rolled in γ -zone and then cooled in α -zone, during which $\langle 100 \rangle$ //ND oriented grains grow. The purity of Fe is particularly important in the invention. When the purity is less than 99.95 mass%, $\langle 100 \rangle$ //ND oriented grains hardly grow in the cooling. Therefore, Fe is not less than 99.95 mass%, preferably not less than 99.98 mass%.

.C+N+S: not more than 10 mass ppm, O: not more than 50 mass ppm.

[0015] These gas components in the pure iron form carbide, oxide and the like with metallic elements (Al, Ti, Nb, Mn and the like) contained at extremely slight amounts of few - few tens mass ppm in the pure iron to obstruct occurrence and growth of nucleus for $\langle 100 \rangle$ //ND oriented grains. And also, the corrosion of pure iron based material is mainly caused by starting from C, N, S segregated in a grain boundary or oxides existing in the grain boundary or in the grains to create rust.

[0016] Such a bad influence of C, N, S and O appears even when C+N+S exceeds 10 mass ppm or even when O exceeds 50 mass ppm, so that it is necessary to satisfy C+N+S: not more than 10 mass ppm and O: not more than 50 mass ppm together. Moreover, preferable content ranges are C+N+S: not more than 5 mass ppm and O: not more than 20 mass ppm.

[0017] Next, production conditions of the pure iron based electromagnetic steel sheet according to the invention are described.

.Hot rolling

[0018] When the raw material of pure iron based steel having the above component composition is hot rolled in α -zone, crystal grains are fined and $\langle 100 \rangle$ //ND oriented grains do not quite grow. Therefore, it is necessary that the hot rolling is carried out at a temperature of γ -zone. When the friction coefficient between a roll and the raw material exceeds 0.3 in the rolling of γ -zone, $\langle 100 \rangle$ //ND oriented grains are apt to be easily generated at a position near to 1/10 of the sheet thickness and hence the occurrence and growth of $\langle 100 \rangle$ //ND oriented grains is controlled. For this end, the hot rolling is carried out at a friction coefficient of not more than 0.3, preferably not more than 0.2. When the rolling under such a condition (so-called lubrication rolling) is conducted in at least one pass of the hot rolling, the effect is developed. Particularly, when it is conducted in a final pass, a larger effect is developed because shearing strain does not concentrate in the surface layer of the steel sheet before transformation. Furthermore, when the strain rate of the rolling is made not less than 150 1/second in the lubrication rolling, the formation of $\langle 100 \rangle$ //ND oriented grains is promoted. Such a tendency is considered to be due to the fact that the formation of oriented grains other than $\langle 100 \rangle$ //ND such as $\langle 110 \rangle$ //ND easily formed on the surface layer portion of the steel sheet or the like is controlled. Moreover, when the strain rate is made not less than 200 1/second, a further(larger)effect is obtained.

[0019] In the above hot rolling in the γ -zone, the total rolling reduction is required to be not less than 50%. Because, when the total rolling reduction in the hot rolling of γ -zone is not less than 50%, the recrystallization in the hot rolling

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is promoted to fine γ -grain size and the $\langle 100 \rangle$ //ND oriented grains are preferentially grown in a direction of sheet thickness in the cooling course after $\gamma \rightarrow \alpha$ transformation. When the total rolling reduction is less than 50%, equiaxed crystal grains having a random direction remain in a central portion of the sheet thickness to degrade the magnetic properties.

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.Cooling after hot rolling

[0020] The $\langle 100 \rangle$ //ND oriented grains in the super-high purity iron grow from the surface of the steel sheet toward a center thereof at α -zone after $\gamma \rightarrow \alpha$ transformation while eroding α -grains newly created through transformation. In this case, when the cooling rate over A_{r3} -300°C exceeds 150°C/min, the grain growing rate does not follow the cooling rate and equiaxed grains remain in the central portion of the sheet thickness. On the other hand, when the cooling rate is slower than 0.5°C/min, the $\langle 100 \rangle$ //ND oriented grains are coarsened, bringing about a degradation of the magnetic properties. Therefore, the cooling rate within a temperature range of A_{r3} -300°C after the rolling is required to be 0.5~150°C/min. Moreover, the preferable cooling rate is 1.0~100°C/min.

[0021] As mentioned above, according to the invention, the effect is first developed by using the pure iron based steel as a raw material and carrying out the production under given conditions, but if any one of the conditions is not satisfied, the gathering degree of $\langle 100 \rangle$ //ND oriented grains can not be enhanced. Moreover, the corrosion resistance is not substantially affected by the production conditions and is dependent upon the component composition.

20 EXAMPLE

[0022] The invention is concretely described with respect to examples.

[0023] A pure iron based steel having a chemical composition shown in Table 1 is melted in a melting furnace of super-high vacuum (10^{-8} Torr) provided with a water-cooled type copper crucible to form an ingot of 10 kg. The ingot is hot forged in γ -zone to form a rod-shaped raw material of 25 mm in thickness. The rod-shaped raw material is heated to 1100°C and hot rolled to a sheet thickness of 1 mm (partly thickness of 5 mm and 13 mm). In this case, the hot rolling is carried out by changing the friction coefficient between the roll and the raw material, and strain rate and the like in the final pass. Further, the cooling rate after the rolling is varied within a wide range. These production conditions are shown in Table 2.

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Table 1

Steel	Fe/mass %	C/mass ppm	N/mass ppm	S/mass ppm	C+N+S/mass ppm	O/mass ppm	A_{r3} transformation point (°C)	Remarks
A	99.99	0.2	0.5	1.2	1.9	21	908	Example
B	99.98	1.1	1.3	1.7	4.1	18	905	Example
C	99.96	2.1	1.9	4.3	8.3	33	900	Example
D	99.97	8.4	9.2	12.1	<u>29.7</u>	28	898	Comparative Example
E	99.96	3.1	2.7	4.1	9.9	<u>80</u>	900	Comparative Example
F	<u>99.91</u>	4.2	2.3	3.1	9.6	16	895	Comparative Example

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Table 2

No	Steel	Final pass of hot rolling			Total rolling reduction of hot rolling (%)	Finishing sheet thickness (mm)	Cooling rate over A_{r3} ~300°C (°C/minute)	Diffraction rate of X-ray I_{100}/I_0	Magnetic flux density B50 (T)	Iron loss W15/50 (W/kg)	Corrosion loss (g/m ²)	Remarks
		Temperature of hot rolling (°C)	Friction coefficient	Strain rate (1/second)								
1	A	930	0.1	250	96	1.0	20	53	1.78	1.5	0.55	Example
2	A	930	0.3	210	95	1.0	70	49	1.77	1.6	0.51	Example
3	A	910	0.1	320	96	1.0	1.5	61	1.79	1.4	0.45	Example
4	A	850	0.2	180	96	1.0	20	1.1	1.55	3.3	0.87	Comparative Example
5	A	910	no lubrication (0.6)	180	96	1.0	20	1.6	1.55	3.4	0.96	Comparative Example
6	B	920	0.2	210	96	1.0	40	45	1.76	1.7	0.68	Example
7	B	940	0.2	160	48	13.0	80	1.2	1.52	3.6	0.98	Comparative Example
8	C	920	0.1	260	96	1.0	90	40	1.75	1.7	0.79	Example
9	C	920	0.2	260	96	1.0	0.3	13	1.68	2.1	0.98	Comparative Example
10	C	940	0.2	210	80	5.0	40	21	1.75	1.8	0.98	Example
11	D	920	0.2	240	96	1.0	50	1.3	1.38	8.5	21.4	Comparative Example
12	E	920	0.1	260	96	1.0	50	3.4	1.60	2.9	14.2	Comparative Example
13	F	930	0.3	260	96	1.0	50	1.2	1.53	3.5	36.2	Comparative Example

[0024] The texture of the resulting hot rolled sheet is measured at a position corresponding to 1/4 of the sheet thickness by an X-ray. And also, a test piece of 1.0 mm in thickness is cut out from a central portion of the thickness of the hot rolled sheet and then a ring-shaped specimen having an inner diameter of 50 mm and an outer diameter of 60 mm is punched out therefrom, and thereafter a primary coil and a secondary coil are wound on the specimen every 100 turns to measure magnetic properties. As the magnetic properties, there are adopted a magnetic flux density (B50) when an external magnetic field of 5000 A/m is applied and an iron loss (W15/50) when it is magnetized to 1.5 T in an alternating magnetic field of 50 Hz.

[0025] The corrosion resistance is evaluated by immersing in aqua regia of 20°C (mixed solution of concentrated nitric acid and concentrated hydrochloric acid at a volume ratio of 1:3) for 100 seconds to measure the corrosion rate. It can be said that when the corrosion rate is not more than 1.0 g/m², the corrosion resistance is satisfactory under usual use environment.

[0026] The test results are shown in Table 2. As seen from Table 2, the invention examples are excellent in both magnetic properties and corrosion resistance. On the contrary, the comparative examples are largely poor in at least one of the magnetic properties and the corrosion resistance as compared with the invention examples.

INDUSTRIAL APPLICABILITY

[0027] As mentioned above, according to the invention, it is possible to gather {100} orientation parallel to the sheet surface after the completion of the hot rolling without passing through complicated steps such as decarburization annealing after cold rolling and the like, so that it is possible to cheaply provide hot rolled electromagnetic steel sheets having excellent magnetic properties.

Claims

1. A hot rolled electromagnetic steel sheet **characterised by** consisting of a super-high purity iron comprising Fe: not less than 99.95 mass%, C+N+S: not more than 10 mass ppm, O: not more than 50 mass ppm and the remainder being inevitable impurity, and having excellent magnetic properties and corrosion resistance, wherein the X-ray diffraction ratio I_{100}/I_0 of the steel sheet is not less than 21.
2. A method of producing a hot rolled electromagnetic steel sheet having excellent magnetic properties and corrosion resistance, **characterised in that** the X-ray diffraction ratio I_{100}/I_0 of the steel sheet is not less than 21 and the steel sheet comprises a super-high purity iron comprising Fe: not less than 99.95 mass%, C+N+S: not more than 10 mass ppm, O: not more than 50 mass ppm and the remainder being inevitable impurity and is heated to γ -zone and subjected in this γ -zone to hot rolling at a total rolling reduction of not less than 50% and under condition that at least one pass has a friction coefficient between roll and rolling material of not more than 0.3 and thereafter cooled at an average cooling rate of 0.5~150°C per minute within a temperature range of A_r_3 transformation point ~300°C.
3. A method according to claim 2, wherein in the at least one pass the strain rate is not less than 150 second⁻¹.

Patentansprüche

1. Warmgewalztes elektromagnetisches Stahlblech, **dadurch gekennzeichnet, dass** es aus einem besonders hochreinen Eisen besteht, das enthält: nicht weniger als 99,95 Massenprozent Fe, nicht mehr als 10 Massen-ppm C + N + S, nicht mehr als 50 Massen-ppm O, wobei der Rest aus unvermeidbaren Verunreinigungen besteht und das Stahlblech ausgezeichnete magnetische Eigenschaften und eine ausgezeichnete Korrosionsbeständigkeit aufweist, und das Röntgenstrahlen-Brechungsverhältnis I_{100}/I_0 des Stahlblechs nicht kleiner ist als 21.
2. Verfahren zum Herstellen eines warmgewalzten elektromagnetischen Stahlblechs, das ausgezeichnete magnetische Eigenschaften und eine ausgezeichnete Korrosionsbeständigkeit aufweist, **dadurch gekennzeichnet, dass** das Röntgenstrahlen-Brechungsverhältnis I_{100}/I_0 des Stahlblechs nicht kleiner ist als 21 und das Stahlblech aus einem besonders hochreinen Eisen besteht, das enthält: nicht weniger als 99,95 Massenprozent Fe, nicht mehr als 10 Massen-ppm C + N + S, nicht mehr als 50 Massen-ppm O, wobei der Rest aus unvermeidbaren Verunreinigungen besteht, und das Stahlblech in den γ -Bereich aufgeheizt wird und in diesem γ -Bereich warmgewalzt wird, und zwar mit einer Gesamtwalzreduktion von nicht weniger als 50 Prozent und unter der Bedingung, dass mindestens ein Durchgang einen Reibungskoeffizient zwischen der Walze und dem gewalzten Material von nicht mehr

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als 0,3 aufweist, und das Stahlblech anschließend mit einer mittleren Abkühlrate von 0,5 - 150°C pro Minute innerhalb eines Temperaturbereichs des A_{r3} -300°C-Umwandlungspunkts abgekühlt wird.

- 5 3. Verfahren nach Anspruch 2, wobei im mindestens einen Durchgang die Umformungsgeschwindigkeit nicht kleiner ist als 150 Sekunde⁻¹.

Revendications

10 1. Feuille d'acier électromagnétique laminée à chaud, **caractérisée en ce qu'elle** est constituée en fer de très haute pureté, comprenant Fe : pas moins de 99,95 % en poids, C + N + S : pas plus de 10 ppm en poids, O : pas plus de 50 ppm en poids, le reste étant constitué par d'inévitables impuretés, et présentant des propriétés magnétiques et une résistance à la corrosion excellentes, feuille dans laquelle le rapport de diffraction des rayons X I_{100}/I_0 de la tôle d'acier n'est pas inférieur à 21.

15 2. Procédé de production d'une feuille d'acier électromagnétique laminée à chaud présentant des propriétés magnétiques et une résistance à la corrosion excellentes, **caractérisé en ce que** le rapport de diffraction des rayons X I_{100}/I_0 de la tôle d'acier n'est pas inférieur à 21 et **en ce que** la tôle d'acier contient du fer de très haute pureté, comprenant Fe : pas moins de 99,95 % en poids, C + N + S : pas plus de 10 ppm en poids, O : pas plus de 50 ppm en poids, le reste étant constitué par d'inévitables impuretés, et est chauffée pour passer en phase γ et, dans cette phase γ , est soumise à un laminage à chaud, à une réduction totale, au laminage, qui n'est pas inférieure à 50 % et à condition qu'au moins une passe ait un coefficient de friction entre le rouleau et le matériau laminé n'excédant pas 0,3, puis est ensuite refroidie à un taux moyen de refroidissement de 0,5 - 150°C par minute, dans une gamme de température A_{r3} , point de transformation - 300°C.

25 3. Procédé selon la revendication 2, dans lequel, au cours de la passe qui est au minimum d'une, la vitesse de déformation n'est pas inférieure à 150 secondes⁻¹.