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(54) **METHOD FOR MANUFACTURING NON-ORIENTED SILICON STEEL WITH HIGH-MAGNETIC INDUCTION**

(57) Abstract: A manufacture process of non-oriented silicon Steel with high magnetic induction comprising the procedures: 1) smelting and casting: the steel's chemical compositions by weight percent: Si 0.1~1%, Al 0.005~1.0%, C ≤ 0.004%, Mn = 0.10~1.50%, P ≤ 0.2%, S ≤ 0.005%, N ≤ 0.002, Nb+V+Ti ≤ 0.006%, and the rest is Fe; molten steel is smelted and secondary refined and then casted into a billet; 2) hot-rolling: the billet is heated to 1150~1200°C, and then hot-rolled into a plate at finish-rolling temperature 830~900 °C, at a temperature ≥ 570°C, and is coiled; 3) flattening: the plate is cold-rolled at compression ratio 2~5%; 4) normalization: the plate is

normalized at temperature not below 950°C for 30~180s; 5) pickling and cold-rolling: the normalized plate is pickled, and then is successively cold-rolled several times at total compression ratio 70~80% into a sheet with thickness of finished product; 6) finish-annealing: the cold-rolled sheet is quickly heated to 800~1000 °C at temperature rise rate ≥ 100°C/s, and soaked for 5~60s, thereafter, cooled slowly to 600~750C, then left to cool naturally. The manufacture process can raise magnetic induction of non-oriented silicon Steel by at least 200 Gauss without increasing iron loss.

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Description**Field of the Invention**

5 **[0001]** This invention relates generally to a manufacture process of non-oriented silicon Steel, and particularly, to a manufacture process of non-oriented silicon Steel with high magnetic induction.

Background of the Invention

10 **[0002]** Non-oriented silicon Steel is an important magnetic material and widely used in manufacture of various electric machines, compressors and so on. In general, it contains silicon of less than 6.5%, aluminum of less than 3%, carbon of less than 0.1%, and other trace elements. Manufacture process of silicon Steel includes the procedures of hot-rolling, normalization, cold-rolling, finish-annealing and coating with insulation film.

15 **[0003]** As to non-oriented silicon Steel, main property indexes of include iron loss, magnetic induction and magnetic anisotropy. The magnetic properties of non-oriented silicon Steel are very prone to be affected by various factors such as material compositions, thickness, heat treatment procedure, and so on.

20 **[0004]** In order to obtain non-oriented silicon Steel with superhigh magnetic induction, a common practice is to reduce silicon content and thus to reduce material electric resistivity, meanwhile, to adopt a higher normalization temperature for hot-rolled plate, for example, even up to 1000 °C . However, because the contents of silicon and aluminum are rather lower, the re-crystallized structure of the normalized non-oriented silicon steel plate is quite fine. The fine grain structure generated in normalization will cause the surface texture {0k1} of finish-annealed sheet to have a rather low intensity, and accordingly, a lower magnetic induction.

25 **[0005]** Moreover, anneal procedure is also a critical factor to affect on magnetic induction of silicon Steel. In order to make annealed sheet have appropriate-sized grains, a common practice is to employ an appropriate soakage temperature and an appropriate soakage period. If soakage temperature is too high and soakage period is too long, the crystal grains of annealed silicon Steel will be rather coarse, the surface texture {111} will be intensified, and magnetic induction of the sheet will be weakened; contrarily, if diameters of the grains are on the small side, the hysteresis loss of material will be on the large side, which will increase electric loss in end use.

30 **[0006]** In anneal procedure, as compared to heating at a lower temperature rise rate, heating at a higher temperature rise rate will bring about quite intensive Gauss texture. Whereas, heating at a lower temperature rise rate will result in that the texture of the finished silicon Steel product is composed of more component {111} <112> and less components {110} <114>, {001} <120> and {111} <110>. (see paper: Jong-Tae PARK, Jerzy A.SZPUNAR Sang-Yun CHA Effect of heating Rate on the development of Annealing Texture in Non-oriented Electrical steels ISIJ International, Vol.43(2003), No.10, pp.1611-1614). Therefore, in anneal procedure, heating at a higher temperature rise rate can depress recovery and give a surface texture with {110} and {100} in core, and so that effectively improve magnetic induction of the finished silicon Steel product.

Summary of the Invention

40 **[0007]** The objective of the invention is to provide a manufacture process of non-oriented silicon Steel with high magnetic induction, the manufacture process is characteristic of including the measures: to roll the hot-rolled plate lightly and to heat the cold-rolled sheet quickly to an anneal temperature so as to get non-oriented silicon Steel with high magnetic induction under the precondition of not increasing iron loss of the sheet.

45 **[0008]** In order to attain the above objective, the invention's manufacture process of non-oriented silicon Steel with high magnetic induction comprises the following procedures:

1) Smelting and casting

50 **[0009]** The chemical compositions of the non-oriented silicon Steel, by weight percent, are: Si 0.1~1%, Al 0.005~1.0%, C ≤ 0.004%, Mn = 0.10~1.50%, P ≤ 0.2%, S ≤ 0.005%, N ≤ 0.002, Nb+V+Ti ≤ 0.006%, and the rest is Fe and unavoidable inclusions; the non-oriented silicon Steel is smelted and secondary refining treated in a converter or electric furnace, and then casted into a steel billet;

2) Hot-rolling

55 **[0010]** The steel billet is heated to a temperature between 1150~120°C, and soaked at the temperature for a certain time, and then hot-rolled into a steel plate at finish-rolling temperature of 830~900°C; when being cooled to a temperature ≥ 570°C, the hot-rolled plate is coiled;

3) Flattening

[0011] The hot-rolled plate is cold-rolled at rolling compression ratio of 2~5%;

4) Normalization

[0012] After being cold-rolled, the hot-rolled plate is continuously normalized at one time at a temperature not below 950°C, and maintained at the temperature for 30~180s;

5) Pickling and cold-rolling

[0013] The normalized plate is pickled, and then is successively cold-rolled several times at a progressive or total rolling compression ratio of 70~80% finally into a cold-rolled silicon steel sheet with the thickness of its finished product;

6) Annealing

[0014] The cold-rolled sheet is quickly heated to a temperature between 800~1000°C at a temperature rise rate of not below 100°C/s, and maintained at the temperature for 5-60s, thereafter, cooled slowly to 600~750 °C at a cooling rate of 3~15°C/s.

[0015] In preferred embodiment, the atmosphere of the Annealing, by volume percent, is H₂ of 30%~70% + N₂ of 70%~30%, the dew point ≤ -25°C.

[0016] The main factors to have effect on magnetic induction intensity B₂₅ and B₅₀ of non-oriented silicon Steel are chemical compositions and crystal grain texture. Higher contents of silicon, aluminum and manganese will result in a higher current resistivity and lower magnetic properties B₂₅ and B₅₀. The ideal crystal texture is surface texture (100) [uvw] because it is isotropic and the hard-magnetized direction is not on the rolled surface. In practice, it is impossible to get a single surface texture of this kind. In general, there exist texture components (100) [011], (111) [112], (110) [001], (112) [011] and so on, of them, texture component (100) only amounts to 20% or so and largely belongs in non-oriented disordered texture, i. e. magnetic anisotropic one. Hereby, to change chemical compositions of material and to improve manufacture process so as to intensify component (100) and to weaken component (111) is an important approach to raise magnetic induction intensity B₂₅ and B₅₀.

[0017] In composition design of the invention, the following points are primarily taken into account:

Si: it is soluble in ferrite to form substitution solid solution so as to increase material resistivity and reduce iron loss, and thus, is the most important alloying element of electric steel, but it is adverse to magnetic induction. The invention is aimed at non-oriented silicon Steel with high magnetic induction, therefore, Si content is determined as low as 0.1~1%.

Al: it is also an element to increase resistivity, and is soluble in ferrite to increase material resistivity and to make crystal grains coarse and to reduce iron loss, but it will also reduce magnetic induction. Al content of more than 1.5% will cause smelting, casting and machining to be difficult and will reduce magnetic induction.

Mn: like Si and Al, it will increase steel's resistivity and reduce magnetic induction, but it is advantageous to reduce iron loss, and it will react with composition S to generate stable MnS so as to eliminate the adverse influence of S on magnetic property. Therefore, it is necessary to have Mn content of over 0.1% in the silicon Steel. In the invention, Mn content is controlled within 0.10~1.50%.

P: to add P of a certain content in steel's compositions can improve manufacturability of silicon Steel, but P content shall be below 0.2%.

C, N, Nb, V and Ti: they are all elements adverse to magnetic property. In the invention, it is controlled that C ≤ 0.004%, S ≤ 0.005%, N ≤ 0.002, Nb+V+Ti ≤ 0.006% so as to minimize their adverse effect on magnetic property.

[0018] Temperature of heated billet or slab shall be below the solid solution temperature of inclusions MnS and AlN in the steel. In the invention, heating temperature is set at 1150~120°C, finish rolling temperature is set at 830~900 °C, and coiling temperature is set not below 570 °C, these temperatures can impede solid solution of the inclusions and make the hot-rolled plate have coarse grains.

[0019] In the invention, to flatten the hot-rolled plate appropriately is a key factor to attain superhigh-magnetic-induction non-oriented silicon Steel. The invention aims at a manufacture process of superhigh-magnetic-induction non-oriented silicon Steel, therefore, the contents of silicon and aluminum in chemical compositions of the steel are controlled to be rather low. However, too small contents of silicon and aluminum will give rise to such a case that crystal grains can not normally grow up in the procedure of normalization of the hot-rolled plate. Moreover, non-oriented silicon steel plate with a lower silicon content trends to generate re-crystallization in the course of being hot-rolled, which will lead to such a

case that there are more fine equiaxed re-crystallized grains and less rolled fiber texture in the metallographic texture of the hot-rolled plate. To flatten the hot-rolled plate at a rolling compression ratio of 2~5% before it is normalized can increase deformation stored energy so as to make the re-crystallized texture of the normalized plate be much coarser. A too high rolling compression ratio in flattening procedure will cause the hot-rolled plate to have more internal defects so as to affect grain growth.

[0020] The intension to have the hot-rolled plate normalized and pre-annealed is to improve grain structure and texture. A research on non-oriented silicon Steel indicates that to make grain structure become coarse prior to cold-rolling can weaken texture component {111} of the cold-rolled sheet and can intensify texture component {0k1} of the cold-rolled sheet after it is finish-annealed, texture component {0k1} is advantageous to magnetic property. Moreover, the incidental phenomenon of separated substance becoming coarser can make grains grow up even easier so as to improve magnetic induction and reduce iron loss. In the invention, normalization temperature of high-magnetic-induction non-oriented silicon steel plate is not below 950°C, soakage period is 30~180s.

[0021] The grains of {110} Gauss texture which are advantageous to magnetic property are usually to nucleate and grow up in the shear-deformed zone of the cold-rolled material. If temperature rise rate is too low, in the phase of temperature being lower, a recovery process in material will occur, which will reduce lattice distortion, thus, the probability of Gauss texture to nucleate will greatly fall down. To use a high temperature rise rate in annealing procedure can rapidly go through the temperature range adverse to evolution of Gauss texture and can make the surface texture {0k1} advantageous to magnetic property evolve even better, and thereby, can optimize magnetic induction and iron loss. To cool the annealed sheet slowly can improve its magnetic property. In the invention, the cold-rolled sheet is finish-annealed by quickly heating to a temperature between 800~1000°C at a temperature rise rate of $\geq 100^\circ\text{C/s}$ and a soakage period of 5~60s, thereafter, is slowly cooled to 600~750°C at cooling rate of 3~15°C /S.

[0022] In comparison to conventional manufacture processes, the manufacture process of the invention can raise magnetic induction of non-oriented silicon Steel by at least 200Gauss under the precondition to maintain the same iron loss.

Brief Description of Drawings

[0023] Fig. 1 shows an interrelation between the compression ratio at which the hot-rolled plate is cold-rolled and the magnetic property of the finish-annealed Steel.

Detailed Description of the Invention

[0024] The invention is now described in detail by embodiments and with reference to the accompanying drawing.

Embodiment 1

[0025]

(1) Hot-rolled non-oriented silicon steel plate with 2.6mm thickness, its compositions and their contents are: Si 0.799%, Al 0.4282%, C 0.0016%, Mn 0.26%, $P \leq 0.022\%$, $S \leq 0.0033\%$, $N \leq 0.0007\%$, Nb 0.0004%, V 0.0016%, Ti 0.0009%, the rest is Fe and unavoidable inclusions.

(2) The hot-rolled plate is cold-rolled at a compression ratio of 1~10%.

(3) The cold-rolled plate is normalized at normalization soakage temperature of 970°C and maintained at the temperature for 60s, thereafter the normalized plate is pickled, and then, cold-rolled into a Steel of 0.5mm thickness.

(4) the cold-rolled sheet is annealed at a high heating rate in an electric annealing furnace in a laboratory, with temperature rise rate of 250°C/s, soakage temperature of 850°C and soakage time of 13s.

[0026] It is found that in the case of the hot-rolled plate being cold-rolled at a compression ratio of 1~10%, the re-crystallized grains of the normalized sheet after being normalized become clearly much coarser, but the microstructure of the finished silicon Steel product is unchanged greatly. In the case of compression ratio of 4~6%, the magnetic property of the finished silicon Steel product comes to the best with magnetic induction B50 up to 1.83T. Magnetic property of the finish-annealed silicon Steel is shown in Table 1. The interrelation between the compression ratio at which the hot-rolled plate is successively cold-rolled several times into a Steel and the magnetic property of the finish-annealed Steel is shown in Fig 1.

Table 1 magnetic property of finish-annealed non-oriented silicon Steel

Compression ratio	Normalization procedure	Anneal procedure	P15/50	B50
0	970°C for 60s	850°C for 13s	4.495	1.813
1%			4.392	1.816
2%			4.245	1.827
4%			3.971	1.83
6%			3.982	1.829
8%			3.871	1.823
10%			4.092	1.821

[0027] The microstructures of both the normalized plate and the finish-annealed sheet obtained at different rolling compression ratios are inspected. It is found that after the hot-rolled plate is lightly cold-rolled, the crystal grains of the normalized plate grow up obviously, but the sizes of crystal grains of the finish-annealed sheet are not changed clearly. The mean grain diameters of both the normalized plate and the finish-annealed sheet are shown in Table 2. There is a good corresponding relation between this result and the magnetic property of the finished sheet product. That is, as the grains of the normalized plate become bigger, the texture component {111} of the cold-rolled sheet after being finish-annealed is weakened, while the texture component {110} which is advantageous to magnetic property is intensified, thereby, the magnetic induction B50 of the finish-annealed sheet is optimized.

Table 2 mean grain diameters of both normalized plate and finish-annealed sheet of non-oriented silicon steel

Compression ratio	Normalization procedure	Finish-anneal procedure	Grain diameter of normalized plate, μm	Grain diameter of finish-annealed sheet, μm
0	970°C for 60s	850°C for 13s	65	38
1%			74	40
2%			200	40
4%			288	42
6%			230	40
8%			170	40
10%			170	40

Embodiment 2

[0028]

(1) Hot-rolled non-oriented silicon steel plate with 2.6mm thickness, its compositions and their contents are: Si 1%, Al 0.2989%, C 0.0015%, Mn 0.297%, P 0.0572%, S 0.0027%, N 0.0009%, Nb 0.0005%, V 0.0015%, Ti 0.0011%, the rest is Fe and unavoidable inclusions.

(2) The hot-rolled plate is cold-rolled at rolling compression ratio of 4%.

(3) The cold-rolled plate is normalized at normalization soakage temperature of 950 °C and maintained at the temperature for 60s, thereafter, the normalized plate is pickled, and then cold-rolled into a Steel of 0.5mm thickness.

(4) the cold-rolled sheet is annealed at a high heating rate in an electric annealing furnace in a laboratory, with different temperature rise rates of 20°C/s, 150°C/s and 250°C/s, respectively, soakage temperature of 960°C and soakage time of 13s.

[0029] The magnetic property of the finish-annealed sheet is shown in Table 3.

Table 3 magnetic property of the finish-annealed non-oriented silicon Steel

Temperature rise rate, °C/s	Normalization procedure	Anneal procedure	P15/50	B50
20	950°C for 60s	960°C for 13s	4.564	1.775
150			4.180	1.7885
250			4.100	1.790

[0030] As can be seen in Table 3, the iron loss and magnetic induction of the finish-annealed sheet is affected by temperature rise rate. As temperature rise rate is raised, iron loss is reduced and magnetic induction is increased.

Claims

1. A manufacture process of non-oriented silicon Steel with high magnetic induction comprising the following procedures:

1) Smelting and casting

a non-oriented silicon steel has the following chemical compositions by weight percent: Si 0.1~1%, Al 0.005~1.0%, C ≤ 0.004%, Mn 0.10~1.50%, P ≤ 0.2%, S ≤ 0.005%, N ≤ 0.002, Nb+V+Ti ≤ 0.006%, and the rest is Fe and unavoidable inclusions; the non-oriented silicon steel is smelted and secondary refining treated in a converter or electric furnace, and then casted into a steel billet;

2) Hot-rolling

The steel billet is heated to a temperature between 1150~1200°C, and soaked at the temperature for a certain time, and then hot-rolled into a steel plate at finish-rolling temperature of 830~900°C; when being cooled to a temperature ≥ 570°C, the plate is coiled;

3) Flattening

The hot-rolled plate is cold-rolled at rolling compression ratio of 2~5%;

4) Normalization

After being cold-rolled, the hot-rolled plate is continuously normalized at one time at a temperature not below 950°C, and maintained at the temperature for 30~80s;

5) Pickling and cold-rolling

The normalized plate is pickled, and then is cold-rolled several times into a cold-rolled sheet with the thickness of the finished product at a total rolling compression ratio of 70~80%;

6) Annealing

The cold-rolled sheet is quickly heated-annealed, wherein the temperature rise rate is not below 100°C/s, the temperature is rose to between 800~1000 °C, and maintained at the temperature for 5~60s, thereafter, cooled slowly to 600~750°C at a cooling rate of 3~15°C/s.

2. The manufacture process of non-oriented silicon Steel with high magnetic induction as defined in claim 1, **characterized in that** the atmosphere of the annealing, by volume percent, is H₂ of 30%~70% + N₂ of 70%~30%, and the dew point ≤ -25°C.

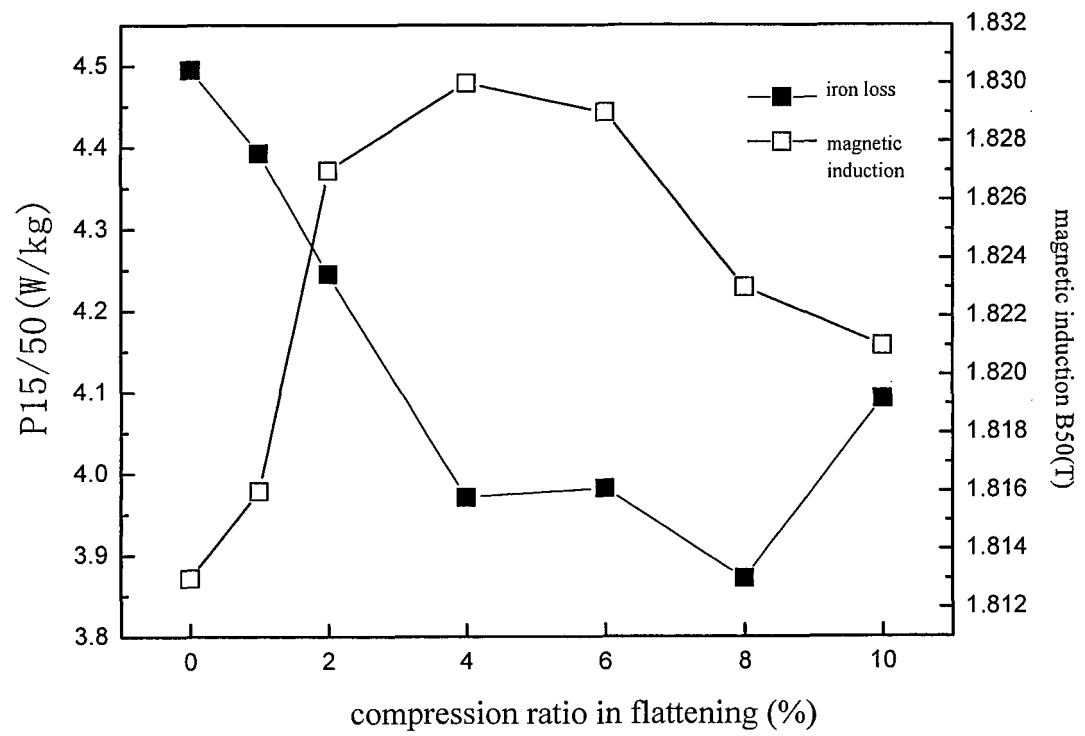


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2011/072775

A. CLASSIFICATION OF SUBJECT MATTER

See extra sheet.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C21D; C22C; H01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI; EPODOC; CPRS; CNKI; non, oriented, electromagnetic, C, carbon, Si, silicon, Mn, manganese, hot, roll, cold, level, normalizing, anneal.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CN 101041222 A (BAOSHAN IRON & STEEL CO., LTD.) 26 Sep. 2007 (26.09.2007) page 3, lines 1~23, page 5, lines 2~7	1-2
A	CN 101343683 A (SHOUGANG CORP.) 14 Jan. 2009 (14.01.2009) the whole document	1-2
A	US 4946519 A (KAWASAKI STEEL CORPORATION) 07 Aug. 1990 (07.08.1990) the whole document	1-2

☐ Further documents are listed in the continuation of Box C.
 ☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier application or patent but published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim (S) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&" document member of the same patent family
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"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 08 July 2011(08.07.2011)	Date of mailing of the international search report 11 Aug. 2011 (11.08.2011)
Name and mailing address of the ISA/CN The State Intellectual Property Office, the P.R.China 6 Xitucheng Rd., Jimen Bridge, Haidian District, Beijing, China 100088 Facsimile No. 86-10-62019451	Authorized officer FU, Xiaoliang Telephone No. (86-10) 82245187

Form PCT/ISA /210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2011/072775

Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
CN 101041222 A	26.09.2007	CN 100546762 C	07.10.2009
CN 101343683 A	14.01.2009	None	
US 4946519 A	07.08.1990	JP 63317627 A	26.12.1988
		US 5013372 A	07.05.1991
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Form PCT/ISA /210 (patent family annex) (July 2009)

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International application No.

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CLASSIFICATION OF SUBJECT MATTER

G21D 8/12 (2006.01) i

G22C 38/02 (2006.01) i

G22C 38/06 (2006.01) i

REFERENCES CITED IN THE DESCRIPTION

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Non-patent literature cited in the description

- **JONG-TAE PARK ; JERZY A.SZPUNAR ; SANG-YUN CHA.** *Effect of heating Rate on the development of Annealing Texture in Non-oriented Electrical steels ISIJ International*, 2003, vol. 43 (10), 1611-1614 [0006]