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(71) Applicant: Baoshan Iron & Steel Co., Ltd. Shanghai 201900 (CN)

(72) Inventor: CHEN, Lingfeng Shanghai 201900 (CN)

 (74) Representative: Jennings, Nigel Robin et al Kilburn & Strode LLP
 20 Red Lion Street London WC1R 4PJ (GB)

(54) METHOD FOR IMPROVING SURFACE COARSE GRAIN OF NON-ORIENTED SILICON STEEL

(57) A method for fining coarse crystal grains at surface of non-oriented silicon , comprising the following steps: 1) smelting and casting; compositions of non-oriented silicon steel by weight percent are: C: $0.001\%\sim0.005\%$, Si: $0.1\%\sim1.8\%$, Mn: $0.10\%\sim0.80\%$, P $\leq 0.04\%$, Al: $0.20\%\sim0.80\%$, S $\leq 0.005\%$, N $\leq 0.005\%$, and the rest is Fe and minimal unavoidable inclusions; molten steel in accordance with the above compositions undergoing smelting and RH refining treatment and then casted into steel billets; 2) hot-rolling into steel sheets; 3)

normalizing, normalizing temperature is controlled at 800~900°C, normalization soaking period is controlled at 15~30S, oxygen content in normalization oven is controlled at 0.5% or less, a ratio of maximum grain size to average grain size in the normalized steel sheets is controlled below 3; and 4) pickling, cold-rolling, annealing, coating in order to obtain non-oriented silicon steel products. The invention, under existing conditions, might fine the coarse crystal grains at the surface of non-oriented silicon steel, without addition of heat treatment procedure and without a parallel hot-working.

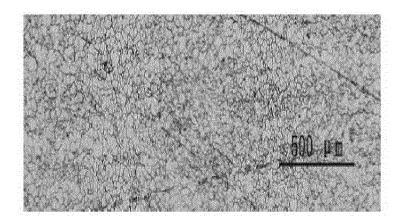


Figure 2

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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 method for fining coarse crystal grains at surface of non-oriented silicon steel.

BACKGROUND

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[0002] The composition contents of existing non-oriented silicon steel products are: C ≤0.005%, Si: 0.1%~1.8%, Mn: 0.10%~0.80%, P: 0.04% or less, A1: 0.20%~0.80%, S ≤0.005%, N ≤0.005%, the rest is Fe and unavoidable inclusions. The above compositions of molten steel are obtained through smelting in a converter and RH refining treatment. After the molten steel is casted into billets and the billets are successively hot-rolled, normalized, pickled, cold-rolled, annealed and coated, products of non-oriented silicon steel are then obtained. Surface quality of the products is often poor, as shown by Figure 1, which have severe coarse crystal grains at the surface.

[0003] At present, solutions to solve the problem of coarse crystal grains of steel products are mainly as follows:

[0004] Chinese patent CN1073982 disclosed a pre-treatment process of "duplex preheating and normalizing" for forgings, which solves the problems that the existing process is incapable of fully fining crystal grains and of clearly improving coarse grains and mixed grains. It includes preheat and normalization procedures, features of which lie in that forgings are preheated to a temperature of 600~710 prior to being normalized. Features of the process lie in that (1) coarse crystal grains are fined; (2) coarse grains and mixed grains are fined to meet relevant technical specifications; and (3) it can be implemented by using existing devices. However, this process might be applied to preliminary heat treatment of large-scale forgings. If coiled sheets of non-oriented silicon steel are subject to this process, an additional heat treatment procedure would be required and cost would be increased.

[0005] Chinese patent CN1804056 disclosed "a method for preventing low-multiple coarse crystal grains of transformable high temperature alloys", which includes two groups of measures. The first group is preventive measures of forging process, which conducts computerized simulation by use of commercial simulation software DEFORM2D so as to determine the deformation in the min-deformable portion of a high temperature alloy forging, and to strictly control the deformation caused by recrystallization generated in the effectively deformed portions in a single heating. The second group is preventive measures of preparative heat treatment, which strictly hold heating temperature for smithing below 1160□, the measures in the second group are used when the measures in the first group do not work successfully or some accidents occur. The production process developed by this invention can make low-multiple coarse crystal grains of the products manufactured of transformable high temperature alloys attain to a qualified grade, and is mainly used for hydraulic pressing deformation and hammer smithing deformation of conventional high temperature alloys. It is not suitable for normalization treatment of non-oriented silicon steel sheets because coiled sheets of non-oriented silicon steel cannot be heat-treated to be deformed by pressing or smithing while being normalized.

[0006] Chinese patent CN1733946 disclosed a treatment process of fine crystal grains of a screw bolt steel used in a sub-critical steam turbine", features of which lie in adding a heat treatment procedure prior to quenching and tempering treatment of materials. The process includes steps of: Step 1: the material is heated to $920\pm20\Box$, which is then maintained for $0.5\sim2$ hours; Step 2: the material is slowly cooled down to $750\pm30\Box$ at a cooling rate of $100\pm20\Box$ per hour, which is then maintained for $0.5\sim2$ hours; Step 3: the material is air-cooled to room temperature. This invention adds a heat treatment prior to quenching and tempering treatment to steel 20Cr1Mo1VNbTiB, to make texture of the material homogenized before being quenching and tempering treated. The material can obtain complete fine crystal grain texture after being quenching and tempering treated, so that defects of coarse crystal grains of steel 20Cr1Mo1VNbTiB can be solved. However, the process cannot solve coarse crystal grains problem that generate in the normalization process of non-oriented silicon steel.

[0007] The above-mentioned three methods can be summed up into two ideas: one is to fine crystal grains so as to eliminate coarse crystal grain through twice heat treatment; the other is to impose critical forging pressing deformation to control recrystallization while the material is being heat-treated, and thereby to solve the problem of low-multiple coarse crystal grains.

[0008] However, the above three methods are not suitable for normalized non-oriented silicon steel products, and the main reason is that the non-oriented silicon steel products can not be hot worked to be deformed while being normalized; if of the twice heat treatment to fine grain size of crystal grains is performed, cost will rise.

55 SUMMARY

[0009] The object of the invention is to provide a method for fining coarse crystal grains at surface of non-oriented silicon steel. This method, under existing conditions, might fine the coarse crystal grains at the surface of non-oriented

silicon steel without addition of heat treatment procedure and without a parallel hot-working, and might have surface quality of the non-oriented silicon steel meeting relevant requirements, without any influence on electromagnetic property of non-oriented silicon steel.

[0010] In order to attain the object above, the method provided by the invention is:

[0011] 1) smelting and casting:

[0012] compositions of a non-oriented silicon steel, by weight percent are: C: $0.001\% \sim 0.005\%$, Si: $0.1\% \sim 1.8\%$, Mn: $0.10\% \sim 0.80\%$, P $\leq 0.04\%$, A1: $0.20\% \sim 0.80\%$, S $\leq 0.005\%$, N ≤ 0.005 , and the rest being Fe and unavoidable inclusions;

[0013] molten steel in accordance with the above compositions is smelted, RH refining treated, and then casted into a steel billet;

[0014] 2) hot-rolling into steel sheets;

[0015] 3) normalizing:

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[0016] normalizing temperature is controlled at 800~900°C, normalization soaking period is controlled at 15~30S, oxygen content in normalization furnace is controlled at 0.5% or less, a ratio of maximum grain size to average grain size in the normalized steel sheet is controlled below 3; and

[0017] 4) pickling, cold-rolling, annealing, coating to obtain a non-oriented silicon steel product.

[0018] Furthermore, the ratio of the maximum grain size to average grain size in the normalized steel sheet is controlled below 2.

[0019] Directing to the coarse crystal grains at the surface of the non-oriented silicon steel product, the invention normalizes the steel sheet, wherein normalizing temperature is controlled at 800~900°C, and normalization soaking period is controlled at 15~30S. If the normalizing temperature is too high and the soaking period is too long, crystal grains will unusually grow up, severe coarse crystal grains defect will occur after cold-rolling and annealing process. Contrarily, if the normalizing temperature is too low and the soaking period is too short, then the post-rolled deformed texture caused by rolling cannot re-crystallize into fine grains, and so corrugation-like defects will occur, which simultaneously deteriorates magnetic induction property. That is, there is a critical normalizing temperature range and a critical normalization period in process of normalizing treatment of the non-oriented silicon steel with the above compositions and having undergone the above ante-normalization treatments. It will cause crystal grains unusually to grow up and then generate coarse crystal grains at surfaces of the steel sheet when the critical normalizing temperature range and the critical normalization period is exceeded or unreached.

[0020] The ratio of maximum grain size to average grain size in the normalized steel sheet shall be controlled less than 3. If this ratio is too high, it trends to cause coarse crystal grains to generate at the surfaces. Preferably, this ratio is controlled less than 2.

[0021] Oxygen content in the normalization furnace shall be controlled below 0.5%. Excessive oxygen content will result in increment of surface oxide layer, which increases difficulty for pickling and influences surface quality.

[0022] Beneficial effects of the invention are:

[0023] 1) the invention does not utilize twice heat treatments, so that operation of the invention is simple and t energy-saving:

[0024] 2) the invention can effectively improve surface quality of the non-oriented silicon steel sheet by the normalizing process, so as to effectively eliminate defects of coarse crystal grains at surface of the non-oriented silicon steel products.

40 BRIEF DESCRIPTION OF THE DRAWINGS

[0025] Fig. 1 is a metallographic photo of coarse crystal grains at the surface of a finished steel product as a comparative object; and

[0026] Fig. 2 is a metallographic photo of coarse crystal grains at the surface of a finished non-oriented silicon steel product of an embodiment of the invention.

DETAILED DESCRIPTION

[0027] The invention is now described in detail by embodiments and in reference to the accompanying drawings.

[0028] The chemical compositions of embodiments of the invention and comparative objects are listed in Table 1, while normalizing parameters are listed in Table 2. After molten steel is smelted in a converter, RH refining treated and then casted into a billet, the billet is successively hot-rolled into slabs, normalized, pickled, cold-rolled, annealed and coated and finally made into products of the non-oriented electric silicon steel. In this process, the slabs are hot-rolled into steel strips of 2.6mm; the steel strips of 2.6mm thickness are normalized; the normalized steel strips are cold-rolled into steel sheets of 0.5mm; the sheets of 0.5mm are then final-annealed and coated. The temperature of the sheets in the final-annealing procedure after cold-rolling procedure is 820°C, annealing period is controlled at 13~15S; and then cold-rolled electromagnetic steel sheets are obtained. Figs 1 and 2 show metallographic textures at the surface of a steel product as a comparative object and at the surface of the cold-rolled non-oriented silicon steel sheet, respectively.

Table 1

							by weight	percent
	С	Si	Mn	Al	S	Р	N	Fe
Embodiment 1	0.005	0.250	0.250	0.20	0.003	0.04	0.003	rest
Embodiment 2	0.003	0.760	0.410	0.39	0.004	0.03	0.002	rest
Embodiment 3	0.004	1.210	0.590	0.61	0.002	0.04	0.003	rest
Embodiment 4	0.003	1.760	0.790	0.78	0.003	0.02	0.004	rest
Embodiment 5	0.002	0.270	0.430	0.59	0.004	0.01	0.005	rest
Embodiment 6	0.003	0.710	0.220	0.76	0.005	0.04	0.002	rest
Embodiment 7	0.004	1.260	0.780	0.22	0.005	0.03	0.003	rest
Embodiment 8	0.001	1.740	0.610	0.42	0.001	0.02	0.004	rest
Comparative 1	0.001	0.240	0.220	0.26	0.004	0.04	0.002	rest
Comparative 2	0.006	0.730	0.430	0.41	0.005	0.02	0.005	rest
Comparative 3	0.005	1.240	0.580	0.63	0.002	0.01	0.004	rest
Comparative 4	0.003	1.780	0.760	0.79	0.003	0.04	0.003	rest
Comparative 5	0.002	0.260	0.420	0.54	0.006	0.06	0.005	rest
Comparative 6	0.004	1.770	0.220	0.79	0.001	0.03	0.001	rest

Table 2

Table 2					
	Normalizing temperature (°C)	Normalization period (S)	Ratio of maximum grain size to average grain size in normalized steel sheets		
Embodiment 1	900	20	1.86		
Embodiment 2	880	20	1.49		
Embodiment 3	850	20	1.25		
Embodiment 4	830	20	1.10		
Embodiment 5	900	30	2.15		
Embodiment 6	880	30	1.94		
Embodiment 7	850	30	1.41		
Embodiment 8	830	30	1.13		
Comparative 1	1000	60	8.5		
Comparative 2	980	60	8.3		
Comparative 3	970	40	7.8		
Comparative 4	950	40	6.3		
Comparative 5	980	50	7.2		
Comparative 6	990	50	6.1		

[0029] As can be seen from Table 2 and Figs 1 and 2, the surface qualities of finished steel sheets obtained from the embodiments of the invention are obviously better than those of the comparative objects, the finished steel sheet products of the invention have got rid of the defects of coarse crystal grains.

Claims

- 1. A method for fining coarse crystal grains at surface of non-oriented silicon steel, comprising the following steps:
 - 1) smelting and casting

compositions of non-oriented silicon steel, by weight percent are: C: $0.001\%\sim0.005\%$, Si: $0.1\%\sim1.8\%$, Mn: $0.10\%\sim0.80\%$, P $\leq 0.04\%$, Al: $0.20\%\sim0.80\%$, S $\leq 0.005\%$, N <0.005%, and the rest is Fe and unavoidable inclusions:

molten steel in accordance with the above compositions is smelted, RH refining treated, and then casted into steel billets;

- 2) hot-rolling into steel sheets;
- 3) normalizing

normalizing temperature is controlled at 800~900°C, normalization soaking period is controlled at 15~30S, oxygen content in normalization furnace is controlled at 0.5% or less, a ratio of maximum grain size to average grain size in the normalized steel sheets is controlled below 3; and

- 4) pickling, cold-rolling, annealing and coating in order to obtain a non-oriented silicon steel product.
- 2. The method for fining coarse crystal grains at surface of non-oriented silicon steel as defined in claim 1, **characterized** in that the ratio of maximum grain size to average grain size in the normalized steel sheets is controlled below 2.

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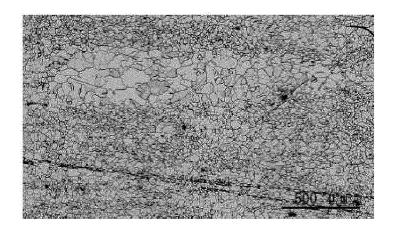


Figure 1

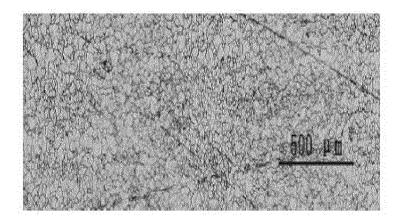


Figure 2

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2011/073358

A. CLASSIFICATION OF SUBJECT MATTER See extra sheet According to International Patent Classification (IPC) or to both national classification and IPC FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC: C21D8/-, C22C38/-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, CN-PAT, CNKI: C, carbon, Si, silicon, silicium, Al, aluminium, aluminum, Mn, manganese, Fe, iron, ferrum, normalizing, grain, texture, hot roll+, cold roll+, non oriented, non directional, electrical steel, electric steel, magnetic steel, silicon steel, si steel C. DOCUMENTS CONSIDERED TO BE RELEVANT Category* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. CN101041222A (BAOSHAN IRON & STEEL CO LTD)26 Sep.2007(26.09.2007) Y 1-2 Example 1 CN1063125A (KAWASAKI STEEL CORP)29 Jul.1992(29.07.1992) 1-2 Y 1-2 Page 9 paragraph 2 Y CN101358318A (SHOUGANG CORP)04 Feb.2009(04.02.2009) claim 6 1-2 JP1306523A (KOBE STEEL LTD)11 Dec.1989(11.12.1989) claim 2 Y 1-2 US4204890A (KAWASAKI STEEL CORP)27 May 1980(27.05.1980) whole document 1-2 ☐ Further documents are listed in the continuation of Box C. See patent family annex. Special categories of cited documents: later document published after the international filing date or priority date and not in conflict with the application but document defining the general state of the art which is not cited to understand the principle or theory underlying the considered to be of particular relevance invention document of particular relevance; the claimed invention earlier application or patent but published on or after the cannot be considered novel or cannot be considered to involve international filing date an inventive step when the document is taken alone document which may throw doubts on priority claim (S) or document of particular relevance; the claimed invention which is cited to establish the publication date of another cannot be considered to involve an inventive step when the citation or other special reason (as specified) document is combined with one or more other such documents, such combination being obvious to a person "O" document referring to an oral disclosure, use, exhibition or skilled in the art other means "&"document member of the same patent family document published prior to the international filing date but later than the priority date claimed Date of mailing of the international search report Date of the actual completion of the international search 11 Aug. 2011 (11.08.2011) 15 Jul.2011 (15.07.2011) Name and mailing address of the ISA/CN Authorized officer The State Intellectual Property Office, the P.R.China CHEN Dazhou 6 Xitucheng Rd., Jimen Bridge, Haidian District, Beijing, China 100088

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No. PCT/CN2011/073358

		T	T
Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
CN101041222A	26.09.2007	CN100546762C	07.10.2009
CN1063125A	29.07.1992	EP0490617A2	17.06.1992
		AU8896991A	11.06.1992
		CA2057368A	11.06.1992
		TW198734A	21.01.1993
		JP5171280A	09.07.1993
		EP0490617A3	15.09.1993
		US5413640A	09.05.1995
		JP2500033B2	29.05.1996
		KR940008933B1	28.09.1994
		CA2057368C	24.06.1997
		EP0490617B1	07.07.1999
		DE69131416E	12.08.1999
		CN1034516C	09.04.1997
		AU629489B	01.10.1992
		DE69131416T	13.01.2000
CN101358318A	04.02.2009	CN101358318B	09.03.2011
JP1306523A	523A 11.12.1989		29.03.1996
		JP2116211C	06.12.1996
US4204890A	27.05.1980	BE871923A	01.03.1979
		DE2848867A	17.05.1979
		BR7807224A	12.06.1979
		SE7811436A	18.06.1979
		JP54068717A	02.06.1979
		FR2408657A	13.07.1979
		CA1103567A	23.06.1981
		DE2848867C	01.10.1981
		JP56054370B	25.12.1981
		BR7807424A	24.07.1979
Farm DCT/IS A /210 (material formilla)	(1.1-2000)	JP1109505C	13.08.1982

Form PCT/ISA /210 (patent family annex) (July 2009)

INTERNATIONAL SEARCH REPORT

Information on patent family members

 $\label{eq:problem} \begin{tabular}{ll} International application No. \\ PCT/CN2011/073358 \end{tabular}$

			71/ CN2011/ 019990
Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
		SE445650B	07.07.1986
		FR2408657B	08.06.1979
		FR240803/B	08.00.1979

Form PCT/ISA /210 (patent family annex) (July 2009)

INTERNATIONAL SEARCH REPORT

 $\label{eq:continuous_policy} International application No. $$ PCT/CN2011/073358$$

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C21D8/12 (2006.01)i		
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REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- CN 1073982 [0004]
- CN 1804056 [0005]

CN 1733946 [0006]