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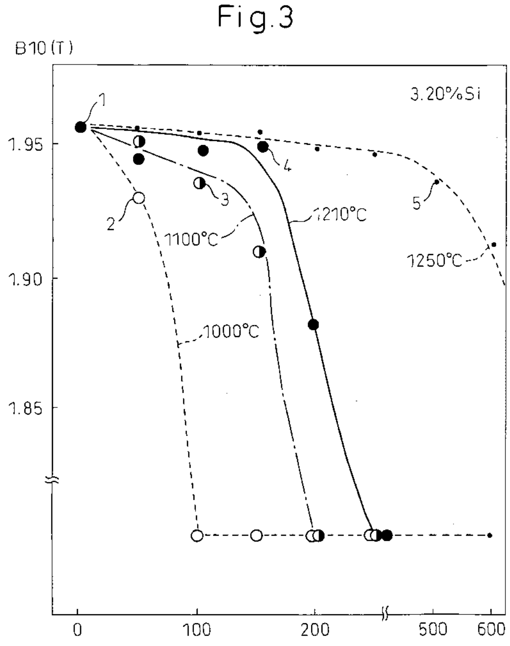
(54) PROCESS FOR MANUFACTURING GRAIN-ORIENTED SILICON STEEL SHEET OF HIGH MAGNETIC FLUX DENSITY

(57) The present invention is a method of using a continuous casting-continuous hot rolling facility to cast a medium thickness bar, making the bar reach a final hot rolling inlet while a temperature over 1200°C for holding the solid-solute state of AlN is maintained, hot rolling the bar, and rapidly cooling it to make fine AlN precipitate and thereby efficiently an inexpensively produce high flux density grain-oriented silicon steel sheet without any high temperature heating of the bar.

Fig.3



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Description

TECHNICAL FIELD

[0001] The present invention relates to a method of production of grain-oriented silicon steel sheet superior in magnetic properties, in particular flux density, used for iron core materials for power transformers, iron core materials for rotating equipment, etc.

BACKGROUND ART

[0002] In the technology of producing grain-oriented silicon steel sheet, as disclosed in U.S. Patent No. 1,965,559, the two-stage cold rolling method of N. P. Goss was developed. The principle of production was clarified as the phenomenon of secondary recrystallization in the presence of fine precipitated MnS by J. E. May and D. Turnbull in 1958 (Trans. AIME., 212 (1958), 769). Separate from this, the inventors developed grain-oriented electrical steel sheet effectively utilizing fine precipitated AlN by the method of single-stage forced cooling with Al (Japanese Patent Publication (B2) No. 33-4710 and U.S. Patent No. 3,159,511). On the other hand, different from precipitates, the fact that fine amounts of solid-solute elements such as Sb, Nb, Se, S, Mo, Cu, and Sn have strong actions inhibiting the growth of primary recrystallized grains and these promote secondary recrystallization was discovered by Saito et al. (Journal of the Japan Institute of Metals, 27 (1963), 191). The secondary recrystallization phenomenon utilized in the current production technology in this way requires the presence of precipitates or fine amounts of solid-solute elements. As a phenomenon of secondary recrystallization in a state with no precipitates at all, the phenomenon of secondary recrystallization in the presence of a base comprised of a stable crystal structure solidified by a certain crystal orientation (Texture Inhibition) was basically clarified by C. G. Dunn (Acta Metallurgica, 1 (1953), 163).

[0003] Furthermore, the principle of production of high flux density grain-oriented silicon steel sheet was elucidated by the inventors by the effect on secondary recrystallization of AlN (Ferrum, Vol. 9, No. 2 (2004), 52)). That is, regarding the effect of AlN on the cold rolling and recrystallization phenomenon of (110)[001]-Goss oriented single crystals, when extremely fine AlN with starting single crystals of 5 nm or less is included in a small amount, the result becomes a {111}<110>-C oriented primary recrystallization growth structure; when fine AlN of 10 nm or so is included in a considerably large amount, the initial orientation (110)[001] is reproduced by secondary recrystallization, and when relatively large AlN of around 1 μm is included in a large amount, similarly secondary recrystallized grains of three types of orientations of the {120}<001>-A orientation, {362}<012>-B orientation, and {111}<110>-C orientation appear due to secondary recrystallization.

[0004] This completely matches with the above-mentioned research results for 3% Si silicon steel single crystals not containing any impurities obtained by C. G. Dunn. From these research results, it is learned that for the production of high flux density grain-oriented silicon steel sheet, securing AlN of around 10 nm in the state of the hot rolled sheet is decisively important. However, with extremely fine AlN of 5 nm or less, secondary recrystallization does not occur, so this should be avoided. Further, coarse AlN of 1 μm or so has no meaning.

[0005] In the past, to secure the AlN of around 10 nm essential for the production of high flux density grain-oriented silicon steel sheet in the state of the hot rolled sheet, the practice had been to reheat a slab obtained by cogging or continuous casting (thickness of about 200 mm or so) to a high temperature of 1300°C or more (flame heating) to make the AlN completely solid-solute, then use a rough rolling mill of a continuous hot rolling facility to obtain a 20 mm to 70 mm thick bar, use a final continuous hot rolling mill to roll this to the final sheet thickness, and coil this around about 500°C, but at the time of finish rolling, the steel was rapidly cooled. However, with a 200 mm or so thickness thick slab, when flame heating at a high temperature of 1350°C or more, the difference in temperature between the slab top surface and bottom surface becomes large, so to obtain the target effect, the need arises to raise the surface temperature extremely high. Therefore, problems in work arise due to the abnormal growth of the slab crystal grains, dissolution of scale on the silicon steel front surface, and buildup in the furnace.

[0006] Solving this problem is extremely difficult, but as one production method, there is the method of employing the low temperature slab heating method (Material Science Forum, 204/206, No. Pt1 (1996), 143) and performing the heating in the final stage after flame heating by induction heating so as to prevent dissolution of the oxide scale (for example, Japanese Patent Publication (A) No. 5-117751) etc. Furthermore, to secure the preferable AlN of around 10 nm by the same thickness as the hot rolled sheet, the idea of employing the several mm thick thin continuous casting method (Japanese Patent Publication (A) No. 2-258922) has also been reported, but the conventional method of cooling a thick CC slab once, then reheating this cooled slab has problems in productivity or work efficiency. Improvement is desired.

[0007] Further, the method of producing a thin slab of a thickness of 20 to 80 mm by continuous casting and employing hot rolling by a rolling start temperature of 1100 to 1200°C to produce a grain-oriented electrical steel sheet is disclosed in Japanese Patent Publication (A) No. 2000-500568. However, with this method, AlN with large grain dimensions is formed during the hot rolling and sufficient magnetic properties cannot be obtained by ordinary treatment processes, so there is the problem that nitridation is required after decarburization annealing.

DISCLOSURE OF THE INVENTION

[0008] As explained above, in the past, the effect of the dispersed precipitation of fine AlN required for production of high flux density grain-oriented silicon steel sheet was achieved by the rapid cooling effect due the high temperature reheating work of a thick slab by a hot rolling soaking furnace making AlN solid-solute in silicon steel, then again hot rolling, but there is a problem due to the dissolution of the scale by the high temperature heating of the thick slab. Further, with the thin continuous casting method for thicknesses of several mm, there are the problem of the crystal orientation and the problem of the embrittlement of the casting structure. There are therefore large problems obstructing practical use.

[0009] Furthermore, with the method of starting the hot rolling of a medium thickness continuously cast slab at a temperature of 1200°C or less, before the hot rolling, AlN precipitates and coarsens and a state sufficiently effective for improving the magnetic properties is not reached.

[0010] The present invention has as its object the provision of a method of production of high flux density grain-oriented silicon steel sheet able to solve the problems in the conventional method of cooling a thick CC slab once, then heating this slab to a high temperature of 1350°C or more, able to greatly improve the work efficiency and energy efficiency, and having a more uniform superior crystal grain orientation and Watt loss than the past by using a continuous casting method to produce a medium thickness slab, holding the slab at a hot rollable temperature of the lowest limit or more, holding the AlN already solid-solute in the state of the melt without causing precipitation until continuous hot rolling, and causing fine precipitation by the rapid cooling effect at the time of continuous hot rolling.

[0011] To achieve this object, the present invention is configured as follows:

(1) A method of production of high flux density grain-oriented silicon steel sheet characterized by continuously casting a melt containing, by mass, C: 0.010 to 0.075%, Si: 2.95 to 4.0%, acid soluble Al: 0.010 to 0.040%, N: 0.0010 to 0.0150%, and one or both of S and Se in 0.005 to 0.1% and having a balance of Fe and unavoidable impurities to produce a medium thickness bar of a thickness of 20 to 70 mm, holding the medium thickness bar at a temperature of over 1200°C after casting, making it reach an inlet of a final hot rolling mill within 500 seconds from the completion of casting to starting continuous hot rolling, hot rolling to obtain a 1.5 mm to 5 mm thick hot rolled sheet, cooling with a cooling time from the end of hot rolling to reaching 600°C in 150 seconds or less, then performing ordinary cold rolling, process annealing, decarburization annealing, final annealing, etc.

(2) A method of production of high flux density grain-oriented silicon steel sheet as set forth in (1), characterized by further including in the melt at least one element selected from the group of Sb: 0.005 to 0.2%, Nb: 0.005 to 0.2%, Mo: 0.003 to 0.1, Cu: 0.02 to 0.2%, and Sn: 0.02 to 0.3% precipitating at grain boundaries to inhibit crystal growth.

(3) A method of production of high flux density grain-oriented silicon steel sheet as set forth in (1), characterized by making the medium thickness bar reach the inlet of the final hot rolling mill within 500 seconds at the longest when holding it at a temperature of 1250°C or more and within 150 seconds when holding it at a temperature of 1200°C or more.

(4) A method of production of high flux density grain-oriented silicon steel sheet as set forth in (1), characterized by heating the medium thickness bar by a heating furnace to hold it at a temperature of 1300 to 1350°C when the time required for making the medium thickness bar produced by continuous casting reach the inlet of the final hot rolling mill and starting the continuous hot rolling is over 200 seconds or when the temperature of the medium thickness bar is a low temperature such as 1000°C.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012]

FIG. 1 is a schematic view of an example of a continuous casting-continuous hot rolling facility.

FIG. 2 is a schematic view of another example of a continuous casting-continuous hot rolling facility.

FIG. 3 is a view showing the effects of the holding temperature and time after AlN solid solution treatment on the magnetic properties (3.20% Si).

FIG. 4 is a view showing typical heat history curves in hot rolling after AlN solid solution treatment (3.10% Si).

FIG. 5 is a view showing the relationship between a rapid cooling (tandem rolling) start temperature and magnetic properties in hot rolling after AlN solid solution treatment (3.10% Si).

FIG. 6 is a view showing the effects of the amount of Si on the cooling curve and precipitation of AlN after AlN solid solution treatment.

BEST MODE FOR CARRYING OUT THE INVENTION

[0013] Below, the present invention will be explained in detail.

[0014] First, the reasons for limitation of the ingredients contained in the molten steel of the present invention will be explained.

[0015] C is an element required for causing a certain γ-transformation during hot rolling in accordance with the amount of Si. If less than 0.010%, it is not possible to stably cause secondary recrystallization. Further, if over 0.075%, the decarburization annealing time becomes longer. This is not preferable for production, so the content was made 0.010 to 0.075%.

[0016] If Si is less than 2.95%, a high grade high flux density grain-oriented silicon steel sheet with a superior Watt loss value is not obtained. Further, if added over 4%, cracking occurs at the time of cold rolling due to embrittlement, so this is not preferred. The content was therefore made 2.95 to 4.0%.

[0017] Acid soluble Al and N are elements required for producing AlN suitable as an inhibitor. The amount sufficient for this purpose was made a range of 0.010 to 0.040% and 0.0010 to 0.0150%.

[0018] S and Se form MnS and MnSe with Mn which act as precipitated dispersed phases for secondary recrystallization. For this purpose, these are included alone or together in an amount of 0.005% to 0.015%. In addition, according to need, to strengthen the inhibitor, it is possible to include at least one element selected from the group of Sb: 0.005 to 0.2%, Nb: 0.005 to 0.2%, Mo: 0.003 to 0.1, Cu: 0.02 to 0.2%, and Sn: 0.02 to 0.3%.

[0019] To produce the high flux density grain-oriented silicon steel sheet of the present invention, it is necessary that 10 nm or so (5 to 50 nm) of AlN be present in the state of the hot rolled sheet. For this reason, continuous casting or another means is used to produce a 20 to 70 mm medium thickness bar, the held heat of this bar or a heat retaining furnace or other heating means for preventing a temperature drop is used to hold the solid solution state of the AlN, while doing so, the bar is made to move to the inlet of the hot rolling mill within a maximum of 150 seconds after extraction from the heat retaining furnace when the temperature is 1200°C or more and within a maximum of 500 seconds when 1250°C or more, the bar is hot rolled to a 1.5 mm to 5 mm thick hot rolled sheet, then the sheet is cooled down to 600°C after the end of the hot rolling within a time of 150 seconds so as to make fine AlN of near 10 nm (5 to 500 nm) precipitate.

[0020] The thickness of the bar in the present invention is limited to a medium thickness of 20 to 70 mm because if less than 20 mm, a large facility is required for heat retention and, further, if over 70 mm, it is not possible to obtain a hot rolled sheet with just a final rolling mill, that is, a rough rolling mill becomes necessary, and economical production is not achieved.

[0021] The means for producing and rolling a 20 to 70 mm thick bar is not particularly limited. An example of a known continuous casting-continuous hot rolling facility is schematically shown in FIG. 1 and FIG. 2. FIG. 1 shows a facility continuously casting a medium thickness slab 2 extracted from a casting mold 1, loading the cut slab 3 in a heat retaining furnace 4 to hold it at a certain temperature, then immediately rolling it by a final continuous hot rolling mill 5 to obtain a thin hot rolled strip steel 6 and coiling it up. Further, FIG. 2 shows a facility continuous casting a medium thickness slab 2, then coiling it into a coil 7, loading the coil into a coil box 8 to even out the temperature, then rolling by a final continuous hot rolling mill 5 and coiling.

[0022] Next, FIG. 3 and FIG. 4 will be used to explain the processing conditions for a medium thickness bar.

[0023] A silicon steel ingot containing, by mass%, 0.045% C, 3.20% Si, 0.025% Al, and a balance of Fe and unavoidable impurities was rolled to obtain a 40 mm thick bar as a starting material. This was divided into four pieces which were tested as follows. These were held in a bar heating furnace 1300°C for 3 hours to make the AlN completely dissolve in the iron metal, then were allowed to cool. When these four types of bars dropped to temperatures of 1250°C, 1210°C, 1100°C, and 1000°C, they were immediately respectively loaded into furnaces held at temperatures of 1250°C, 1210°C, 1100°C, and 1000°C, were respectively held at 1250°C for 480 seconds (total after extraction from bar heating furnace of 500 seconds), at 1210°C for 120 seconds (same, total 150 seconds), at 1100°C for 50 seconds (same, total 100 seconds), and 1000°C for 20 seconds (same, total 50 seconds), then hot rolled and allowed to cool in the air after the end of rolling. The heat histories are shown overall in FIG. 4. In this figure, the curve (A) is the cooling curve in the case of immediately rolling after extraction from the bar heating furnace, while the cooling curves (B), (C), (D), and (E) are as explained above.

[0024] The hot rolled sheets were cold rolled, decarburized, and final annealed to obtain final products, then the products were measured for magnetic properties (B10). The relationship between these properties and the heat histories (total time after extraction from bar heating furnace until reaching final hot rolling mill inlet, including time spent in heat retaining furnace) is shown in FIG. 3.

[0025] As clear from FIG. 3, when performing the hot rolling shown by the cooling curve of FIG. 4(A), the holding time is 0, so the magnetic properties become the most superior B10 properties as shown by the black dots 1 of FIG. 3. The magnetic properties when performing the hot rolling after holding the sheet at 1000°C for 20 seconds of FIG. 4(B), as shown by the white dots 2 of FIG. 3, become considered degraded B10 properties regardless of the holding time being short. If over 100 seconds, the secondary recrystallization itself becomes unstable.

[0026] Furthermore, the magnetic properties when performing the hot rolling after holding the sheet at 1100°C for 50 seconds of FIG. 4(C), as shown by the half black dots 3 of FIG. 3, are improved somewhat due to the long holding time and the high temperature. Furthermore, the magnetic properties when performing the hot rolling after holding the sheet at 1200°C for 120 seconds of FIG. 4(D), as shown by the black dots 4 of FIG. 3, are values close to the best values by making the temperature high even if the holding time is long.

[0027] Finally, the magnetic properties when performing the hot rolling after holding the sheet at 1250°C for 480 seconds of FIG. 4(E), as shown by the small block dots 5 of FIG. 3, are slightly inferior in values compared with the best values regardless of the extremely long holding time.

[0028] In this way, the drop in the bar temperature is critical for the B10 properties, but it is learned that if securing a high temperature over 1200°C, a certain margin can be given to the holding time and superior properties can be obtained.

[0029] When performing the hot rolling shown by the cooling curves of FIGS. 4(B) and (C), as clear from FIG. 3, it is learned that the holding temperatures before rolling are not sufficient and, in such cases, the precipitation of AlN proceeds and a deterioration in the magnetic properties is caused in both cases. Further, in the case of the cooling curve of FIG. 4(C), it is possible to secure a certain extent of magnetic properties if reaching the hot rolling mill inlet in an extremely short time, but if judging from the conditions in the manufacturing site, it is clear that production work is not possible if a heat history along the cooling curve of FIG. 4(D) or FIG. 4(E).

[0030] In the case of 3.0 to 4.0% Si where the Watt loss value of the high flux density grain-oriented silicon steel sheet is emphasized, compared with the case where the amount of Si is a low one of less than 3%, as explained above, the treatment conditions become considerably severe and the time allowed in production work is relatively short. The reason is that in the case of low Si, γ-transformation increases the solid solution degree of the AlN and enables precipitation to be prevented. Therefore, in the case of a high Si, the only option is to utilize the temperature as the means for preventing precipitation. This means that the precipitation of AlN is rapidly retarded the higher the temperature, so when time is required for reaching the inlet of the final hot rolling mill, it may be considered to raise the holding temperature. That is, as shown in claim 4, when the time required for making the medium thickness bar produced by continuous casting reach the inlet of the final hot rolling mill and start the continuous hot rolling exceeds 200 seconds, it is possible to prevent the precipitation of Al by the method of running the bar through a heating furnace held at a temperature of 1250 to 1350°C. Alternative, when the temperature of the bar is a low temperature such as 1000°C, it is possible to prevent the precipitation by the means of running the bar through a heating furnace held at a temperature of 1250 to 1350°C.

[0031] FIG. 5 illustrates the relationship between the magnetic properties and the heat history when rolling a silicon steel ingot comprised of 0.046% C, 3.10% Si, 0.029% Al, and a balance of Fe and unavoidable impurities to produce a 40 mm thick bar, immediately rolling it after heating at 1350°C for 30 minutes to finish it to a 3.5 mm thick hot rolled sheet at about 1000°C, water cooling this from the cooling process right after ending the hot rolling to produce five types of hot rolled sheets, and cold rolling, decarburizing, and final annealing the sheets to produce the final products. In the figure, the thick lines show the starting point of cooling (water cooling) after hot rolling, while the thin lines show the magnetic properties (B10).

[0032] From the results, rapidly cooling the material from as early a timing as possible after the end of hot rolling, that is, cooling in the range up to 150 seconds after the end of hot rolling not by gradual cooling such as a (cooling in the air), but by as fast a rate as possible from a high temperature such as b.c.d.e is necessary to obtain the magnetic properties. For example, in the case of e, a high value of B10=1.95T is obtained. The temperature for cooling in the range not over 150 seconds is made at least 600°C. Normally, a hot rolled steel sheet is coiled up when reaching 600°C or less and is slowly cooled, so AlN no longer precipitates.

[0033] FIG. 6 shows the relationship between the hot rolling and cooling cycle and the amount of precipitation of AlN. For reference, the precipitation curve in the case of a low Si (1.12% Si, 2.17% Si) is simultaneously shown. As will be understood from a comparison with this, when the amount of Si is 3.10%, AlN starts to precipitate from around 1250°C and proceeds rapidly in precipitation at 1200°C or less. In the case of 1.1% Si, AlN does not proceed much in precipitation at all down to 1000°C and first starts to precipitate at 1000°C or less. This is because the α-γ transformation region of the material changes depending on the amounts of C and Si contained and the precipitation behavior of the AlN is closely related to the amount of this γ-transformation.

[0034] Summarizing the above, the hot rolling conditions when utilizing the crystal growth inhibition effect of AlN to produce superior high flux density grain-oriented silicon steel sheet are as follows:

(1) When hot rolling a medium thickness bar comprised of a silicon steel material containing 2.95 to 4% Si in which AlN becomes completely solid-solute, the bar is extracted from the casting or heating furnace, then made to reach an inlet of a final hot rolling mill within a maximum of 500 seconds when the holding temperature is 1250°C or more and preferably within 150 seconds when it is over 1200°C, then started to be hot rolled.

(2) The cooling after the end of the hot rolling is performed so that at the maximum the time until reaching 600°C does not exceed 150 seconds. The AlN precipitates due to the cooling from a high temperature, but if taking time and gradually cooling at this time, the AlN will coarsen along with the elapse of time. In extreme cases, it will become

a size of about 1 μm resulting in a state completely meaningless for the object of the present invention. If the AlN in the completely solid-solute state is cooled to 600°C in a time not exceeding 150 seconds, the precipitated size will become about 10 nm resulting in a state preferable for the present invention.

EXAMPLES

Example 1

[0035] A silicon steel melt comprising, by mass%, 0.045% C, 3.05% Si, and 0.032% Al and having a balance of Fe and unavoidable impurities was cast by a continuous casting machine (hereinafter referred to as a "CC machine") to a 60 mm thick bar. This was immediately hot rolled by final hot rolling to a thickness of 3.0 mm. The final hot rolling inlet temperature at the bar head part was 1210°C and at the tail part was 1205°C. The amount of C of the hot rolled sheet was 0.041% whereby slight decarburization occurred. The sheet was first cold rolled at a reduction rate of 30% to a 2.1 mm thickness, then was annealed at 1100°C for 2 minutes in nitrogen, then cooled by blowing a jet stream over it. The cooling rate was 1100°C to 850°C in about 18 seconds and 850°C to 400°C in about 27 seconds. The AlN after annealing was analyzed as being 0.0055% (NasAlN). Next, the sheet was cooled by a rolling rate of 83.3% to a thickness of 0.35 mm, then decarburized at 800°C for 3 minutes in hydrogen, then annealed at 1200°C for 2.0 hours. The B10 property in the rolling direction of the product was 1.93T, and the W17/50 was 1.15W/kg.

[0036] Comparative Example: A bar of the same ingredients as Example 1 was allowed to stand in front of the final hot rolling mill inlet for about 40 seconds, then started to be final hot rolled. The final rolling start temperature of the bar at that time was 1150°C at bar head part and 1120°C at the tail part. After this, the sheet was treated in the same way as Example 1 and the final product was examined for the secondary recrystallized grain formation rate. This was found to be about 50%, that is, a finished product was not formed.

Example 2

[0037] A silicon steel melt comprising, by mass%, 0.048% C, 3.13% Si, 0.10% Mn, 0.029% Al, and 0.029% S and having a balance of Fe and unavoidable impurities was cast by a CC machine to a 50 mm thick bar. This was immediately hot rolled by final hot rolling to a thickness of 2.8 mm. The final hot rolling inlet temperature at the bar head part was 1210°C and at the tail part was 1200°C. After respectively 10 seconds and 50 seconds, the hot rolling was ended. The temperatures at that time were 1010°C and 1000°C. After about 75 seconds, the coiling was ended. The C after hot rolling was analyzed as being 0.040% and the AlN 0.0040% (NasAlN). This hot rolled sheet was pickled, then cold rolled by a rolling rate of 87.5% to a final gauge of 0.35 mm, was decarburized at 850°C for 3 minutes in wet hydrogen, then annealed in hydrogen at 1200°C for 15 hours. The B10 property in the rolling direction of the product was 1.92T, and the W17/50 was 1.05W/kg.

Example 3

[0038] A silicon steel melt containing, by mass%, 0.050% C, 3.18% Si, 0.075% Mn, 0.021% Al, and 0.035% S and having a balance of Fe and unavoidable impurities was cast by a CC machine to a 40 mm thick bar and immediately rolled by final hot rolling to a thickness of 3.0 mm. The final hot rolling inlet temperature was 1210°C at the bar head part and 1205°C at the tail part. The hot rolling was respectively ended after 12 seconds and after 53 seconds. The temperatures at that time were 1020°C and 990°C. The coiling was completed after about 80 seconds.

[0039] This was continuously annealed at 1100°C for 1 minute in a nitrogen atmosphere, then force cooled by a nitrogen gas spraying system at the outlet of the furnace to 930°C and further rapidly cooled by a laminar blowing system to 200°C. The C at this time was analyzed as being 0.045% and the A1N 0.0040% (NasAlN). The sheet was pickled, then cold rolled by a rolling rate of 88.3% to a 0.35 mm thick final gauge, decarburized at 850°C for 3 minutes in wet hydrogen, then annealed in hydrogen at 1200°C for 15 hours. The magnetic properties in the' rolling direction of the product were a B10 of 1.92T and a W17/50 of 1.05W/kg.

[0040] Comparative Example: A bar of the same ingredients as Example 3 was allowed to stand in front of the final hot rolling mill inlet for about 150 seconds, then started to be final hot rolled. The final rolling start temperature of the bar at that time was 950°C at the bar head part and 930°C at the tail part. After this, the sheet was treated under the same way conditions as Example 3 to obtain the final product, then was examined for the secondary recrystallized grain formation rate. This was found to be about 20%, that is, a finished product was not formed.

Example 4

[0041] A silicon steel melt containing, by mass%, 0.050%C, 3.12% Si, 0.041% Al, 0.030% S, 0.050% Se, and 0.030%

Te and having a balance of Fe and unavoidable impurities was cast by the CC machine to a 60 mm thick bar. This was immediately rolled by final hot rolling to a 3.0 mm thickness. The final hot rolling inlet temperature was 1230°C at the bar head part and 1210°C at the tail part. The hot rolling was ended after 15 seconds and after 60 seconds. The temperatures at this time were respectively 1050°C and 1020°C. The coiling was completed after about 90 seconds.

[0042] The sheet was continuously annealed at 1100°C for 2 minutes in a nitrogen atmosphere, then cold rolled by a rate of 50%, then annealed for 1 minute for primary recrystallization and further rolled by a rolling rate of 84.7% to 0.23 mm. The sheet was annealed by decarburization annealing, then by final annealing at 1200°C for 20 hours along with removal of Se, removal of Te, and removal of S. The magnetic properties of the product were a B10 of 1.93T and a W17/50 of 1.05W/kg.

Example 5

[0043] A silicon steel melt containing, by mass%, 0.046% C, 3.20% Si, 0.031% Al, and 0.025% S and having a balance of Fe and unavoidable impurities was cast by the CC machine to a 50 mm thick bar. This was immediately rolled by final hot rolling to a 2.5 mm thickness. The final hot rolling inlet temperature was 1220°C at the bar head part and 1205°C at the tail part. The hot rolling was ended after 12 seconds and after 50 seconds. The temperatures at this time were respectively 1005°C and 990°C. The coiling was completed after about 85 seconds.

[0044] The sheet was continuously annealed at 1130°C for 2 minutes, then pickled and cold rolled to a final sheet thickness of 0.23 mm, then annealed by decarburization annealing at 850°C for 2 minutes in wet hydrogen. This steel sheet was coated separately with an annealing separator containing, by ratio of weight with respect to MgO: 100, TiO_2: 10 and MnO_2: 5 and furthermore having boric acid added in 0.1 to 3% and with an annealing separator not having boric acid added to it, then was annealed at 1200°C for 20 hours in hydrogen.

[0045] As shown in Table 1, due to the addition of boric acid to this MgO, the B10 property is improved and simultaneously the Watt loss value falls. Further, the variations in them become smaller. The properties of the glass film, very important for grain-oriented silicon steel sheet, become excellent.

Table 1

Amount of addition of boric acid %	Flux density B10 (T)				Watt loss value W17/50 (W/kg)				Remarks Addition of TiO_2, MnO_2
	Lowest	Largest	Average	Difference	Lowest	Largest	Average	Difference	
0	1.88	1.92	1.905	0.04	1.15	1.32	1.235	0.17	No
0.1	1.89	1.94	1.915	0.05	0.99	1.12	1.055	0.13	
0.5	1.90	1.93	1.915	0.03	0.96	1.08	1.020	0.12	Yes
1.0	1.91	1.93	1.920	0.02	0.94	0.98	0.960	0.04	
3.0	1.88	1.91	1.895	0.03	1.02	1.17	1.095	0.15	

Example 6

[0046] A silicon steel melt containing, by mass, 0.04% C, 3.30% Si, and 0.029% Al and having a balance of Fe and unavoidable impurities was cast by the CC machine to a 60 mm thick bar. This was immediately rolled by final hot rolling to a 2.3 mm thickness. The final hot rolling inlet temperature was 1230°C at the bar head part and 1205°C at the tail part. The hot rolling was ended after 12 seconds and after 45 seconds. The temperatures at this time were respectively 1010°C and 995°C. The coiling was completed after about 85 seconds.

[0047] This hot rolled sheet was continuously annealed at 1150°C for 2 minutes, rapidly cooled, pickled, and cold rolled to a final sheet thickness of 0.27 mm and annealed by decarburization annealing at 850°C in hydrogen and by final annealing at 1200°C. It was then cold rolled during and run by the same pass schedule (six passes of 1.6 mm, 1.2 mm, 1.0 mm, 0.8 mm, 0.6 mm, and 0.45 mm) while aging by five different temperatures. That is, the relationship of the conditions and magnetic properties is as shown in Table 2.

[0048] From this, it is learned that aging between passes at 200°C or so is effective.

Table 2

(1)	50°C×5 minutes heat treatment for each cold rolling pass	B10=1.920(T)	W17/50=1.024(W/kg)
(2)	150°C×5 minutes heat treatment for each cold rolling pass	B10=1.944(T)	W17/50=1.001(W/kg)
(3)	200°C×5 minutes heat treatment for each cold rolling pass	B10=1.951(T)	W17/50=0.998(W/kg)
(4)	350°C×5 minutes heat treatment for each cold rolling pass	B10=1.925(T)	W17/50=1.012(W/kg;
(5)	500°C×5 minutes heat treatment for each cold rolling pass	B10=1.880(T)	W17/50=1.195(W/kg)

Example 7

[0049] A silicon steel melt containing, by mass, 0.085% C, 3.20% Si, 0.073% Mn, 0.025% S, 0.025% acid soluble Al, 0.0085% N, 0.08% Sn, and 0.07% Cu and having a balance of Fe and unavoidable impurities was cast by the CC machine to a 60 mm thick bar. This was immediately rolled by final hot rolling to a 2.0 mm thickness. The final hot rolling inlet temperature was 1220°C at the bar head part and 1201°C at the tail part. The hot rolling was ended after 15 seconds and after 55 seconds. The temperatures at this time were respectively 990°C and 985°C. The coiling was completed after about 90 seconds.

[0050] This hot rolled sheet was continuously annealed at 1130°C for 2 minutes, then rapidly cooled in hot water of 100°C and treated by precipitation heat treatment, pickled, then aged at 250°C×5 minutes between passes and cold rolled to a final sheet thickness of 0.22 mm. Next, the sheet was annealed 850°C for 2 minutes in Craced-NH_3 in an atmosphere of a dew point of 62°C by decarburization annealing, was coated by an annealing separator containing a mixture of MgO and TiO_2, and was annealed at 1200°C by final annealing. It was given a tension coating after the final annealing.

[0051] The magnetic properties and crystallinity of the product were B10=1.92(T) and W17/50=0.88W/kg and ASTM No. 5. When Sn and Cu were not added, they were a B10=1.92(T) and W17/5=0.95W/kg and ASTM No. 3.

Example 8

[0052] A silicon steel melt containing, by mass, 00.05% C, 3.05% Si, 0.07% Mn, 0.03% S, and 0.026% acid soluble Al and having a balance of Fe and unavoidable impurities was cast by the CC machine to a 40 mm thick bar. After the casting, the bar was cut. The temperature of the bar at that time was 1255°C. This continued to be held in temperature by a heating apparatus so as not to fall to 1250°C or less, was made to reach an inlet of a final hot rolling mill in about 300 seconds, and immediately started to be hot rolled to a thickness of 30 mm. The final hot rolling inlet temperature was 1220 to 1230°C. The front end and rear end of the hot rolled sheet were finished being hot rolled in 15 seconds and 60 seconds. The temperatures at the time were 1030°C and 1020°C. The sheet finished being coiled after about 70 seconds.

[0053] This hot rolled sheet was continuously annealed at 1130°C for 3 minutes, then force cooled by immersion in a tank filled with boiling water at the outlet of the furnace, pickled, and cold rolled to a 0.3 mm thickness by a 90% reduction rate. This was then annealed by decarburization annealing, then by final annealing at 1200°C for 20 minutes in H_2. The magnetic properties in the rolling direction of the product were B10=1.93(T) and W17/50=1.01W/kg.

[0054] As a comparative example, a bar of the same ingredients as Example 8 was cast, then conveyed to a final hot rolling mill inlet without holding the temperature by a heating apparatus, whereupon the temperature fell to 1100°C. This was immediately rolled by final hot rolling. The hot rolled sheet was treated under the same conditions as Example 3 to obtain a final product. The final product was examined for the secondary recrystallized grain formation rate. This was found to be about 30%, that is, a finished product was not formed.

Example 9

[0055] A silicon steel melt containing, by mass, 0.055% C, 3.20% Si, 0.025% S, and 0.30% acid soluble Al and having a balance of Fe and unavoidable impurities was cast by the CC machine to a 30 mm thick bar. After the casting, the bar was cut. The temperature of the bar at that time was 1150°C. This bar was immediately inserted into a heat furnace heated to 1330°C to make the side AlN solid-solute, then was taken out from the furnace, made to reach the inlet of a final hot rolling mill in about 120 seconds, and immediately started to be hot rolled to a thickness of 25 mm. The final hot rolling inlet temperature was 1210 to 1220°C. The front end and rear end of the hot rolled sheet were finished being hot rolled in 16 seconds and 50 seconds. The temperatures at the time were 1010°C and 998°C. The sheet finished being coiled after about 70 seconds.

[0056] This hot rolled sheet was continuously annealed at 1130°C for 2 minutes, then force cooled by a mist spraying system at the outlet of the furnace, pickled, cold rolled to a 0.3 mm thickness, then annealed by decarburization annealing at 835°C for 3 minutes in wet hydrogen. This steel sheet was coated with a slurry of MgO containing 800 ppm of B, was wound in a coil, and was annealed at 1200°C for 20 hours in hydrogen. The magnetic properties in the rolling direction of the product were B10=1.92(T) and W17/50=0.89W/kg.

[0057] As a comparative example, a bar of the same ingredients as Example 8 was cast, then immediately conveyed to the inlet of a final hot rolling mill. The temperature further dropped and fell to 1100°C. This was immediately rolled by hot rolling. The hot rolled sheet was treated under the same conditions as Example 3 to obtain a final product. The final product was examined for the secondary recrystallized grain formation rate. Just 20% occurred, that is, a finished product was not formed.

INDUSTRIAL APPLICABILITY

[0058] In the present invention, the AlN obtained by rapid cooling at the final hot rolling mill (tandem mill) from the completely solid-solute state in the medium thickness cast slab produced by continuous casting is dispersed uniformly and finely. This is sufficient for producing primary recrystallization nuclei having a superior crystal orientation. Simultaneously, the inhibitory effect on crystal growth is also sufficient. Further, the crystal structure obtained by casting is destroyed by the hot rolling. Therefore, it is possible to obtain high flux density grain-oriented silicon steel sheet forming uniform, complete secondary recrystallized grains by the final annealing and having superior properties of a flux density B10≥1.90T without any detrimental effect of abnormally grown grains of the slab due to conventional high temperature heating. Further, high temperature reheating work of 1350°C by a conventional slab heating furnace is not required at all. The heat held by the steel slab is completely utilized, so leads to remarkable improvement in energy efficiency. The major problem in work due to slab high temperature heating considered difficult in the past can also be solved.

Claims

1. A method of production of high flux density grain-oriented silicon steel sheet **characterized by** continuously casting a melt containing, by mass,

    C: 0.010 to 0.075%,
    Si: 2.95 to 4.0%,
    acid soluble Al: 0.010 to 0.040%,
    N: 0.0010 to 0.0150%, and

    one or both of S and Se in 0.005 to 0.1% and
    having a balance of Fe and unavoidable impurities to produce a medium thickness bar of a thickness of 20 to 70 mm, holding said medium thickness bar at a temperature of over 1200°C after casting, making it reach an inlet of a final hot rolling mill within 500 seconds from the completion of casting to starting continuous hot rolling, hot rolling to obtain a 1.5 mm to 5 mm thick hot rolled sheet, cooling with a cooling time from the end of hot rolling to reaching 600°C in 150 seconds or less, then performing ordinary cold rolling, process annealing, decarburization annealing, final annealing, etc.

2. A method of production of high flux density grain-oriented silicon steel sheet as set forth in claim 1, **characterized by** further including in said melt at least one element selected from the group of Sb: 0.005 to 0.2%, Nb: 0.005 to 0.2%, Mo: 0.003 to 0.1, Cu: 0.02 to 0.2%, and Sn: 0.02 to 0.3% precipitating at grain boundaries to inhibit crystal growth.

3. A method of production of high flux density grain-oriented silicon steel sheet as set forth in claim 1, **characterized by** making said medium thickness bar reach the inlet of the final hot rolling mill within 500 seconds at the longest when holding it at a temperature of 1250°C or more and within 150 seconds when holding it at a temperature of 1200°C or more.

4. A method of production of high flux density grain-oriented silicon steel sheet as set forth in claim 1, **characterized by** heating the medium thickness bar by a heating furnace to hold it at a temperature of 1300 to 1350°C when the time required for making the medium thickness bar produced by continuous casting reach the inlet of the final hot rolling mill and starting the continuous hot rolling is over 200 seconds or when the temperature of said medium thickness bar is a low temperature such as 1000°C.

Fig.1

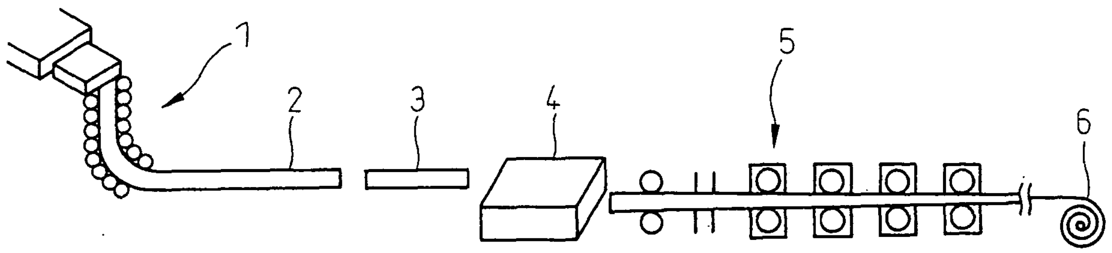


Fig.2

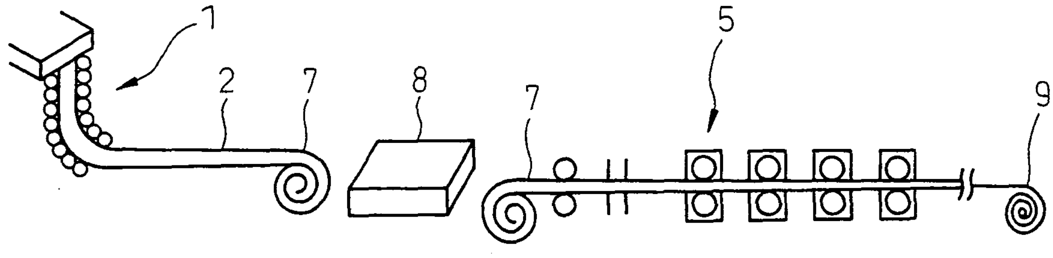


Fig.3

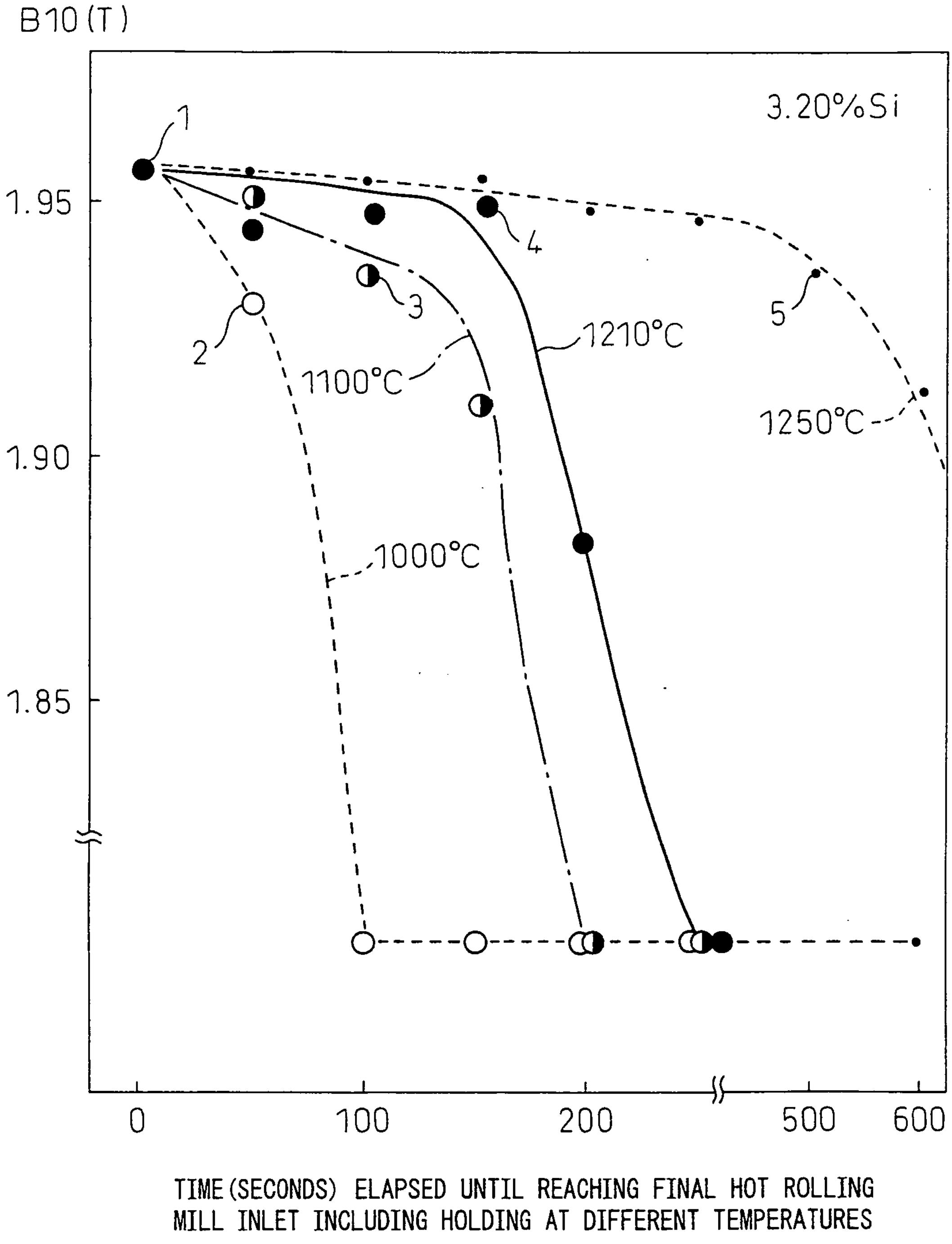

B10 (T)
3.20%Si
1.95
1.90
1.85
1
2
3
4
5
1000°C
1100°C
1210°C
1250°C
0
100
200
500
600
TIME (SECONDS) ELAPSED UNTIL REACHING FINAL HOT ROLLING MILL INLET INCLUDING HOLDING AT DIFFERENT TEMPERATURES

Fig.4

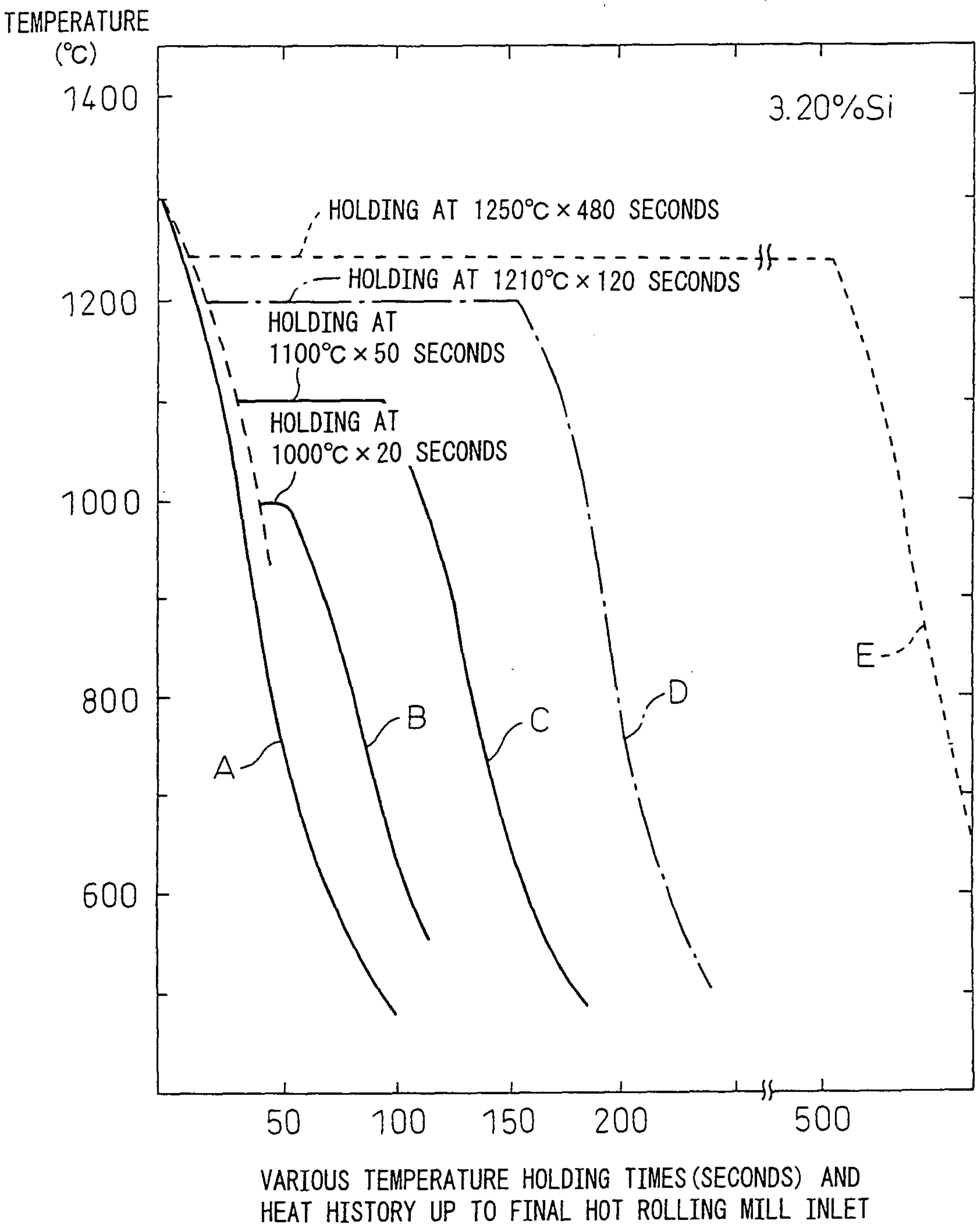

TEMPERATURE (°C)
1400
1200
1000
800
600
3.20%Si
HOLDING AT 1250°C × 480 SECONDS
HOLDING AT 1210°C × 120 SECONDS
HOLDING AT 1100°C × 50 SECONDS
HOLDING AT 1000°C × 20 SECONDS
A
B
C
D
E
50
100
150
200
500
VARIOUS TEMPERATURE HOLDING TIMES (SECONDS) AND HEAT HISTORY UP TO FINAL HOT ROLLING MILL INLET

Fig.5

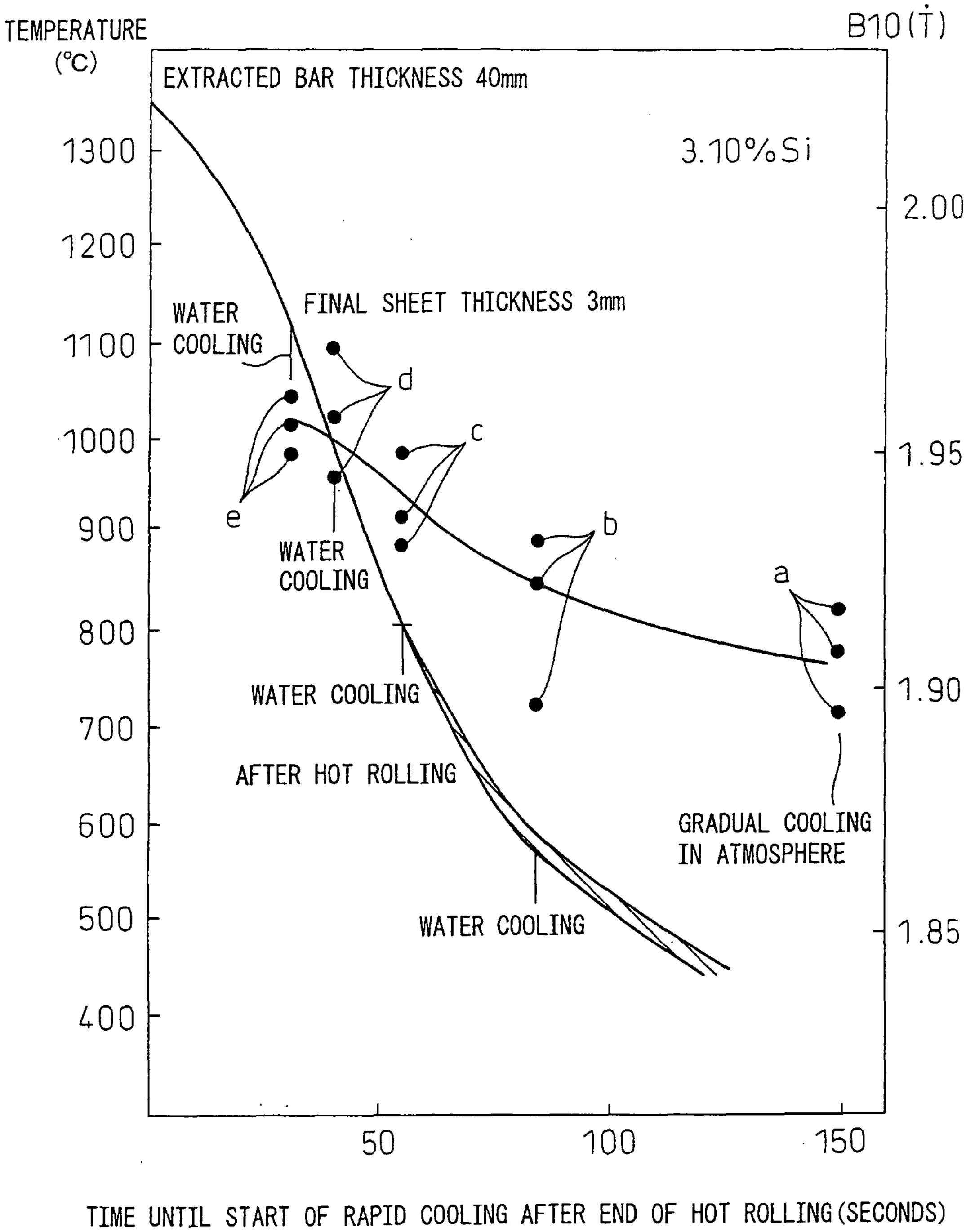
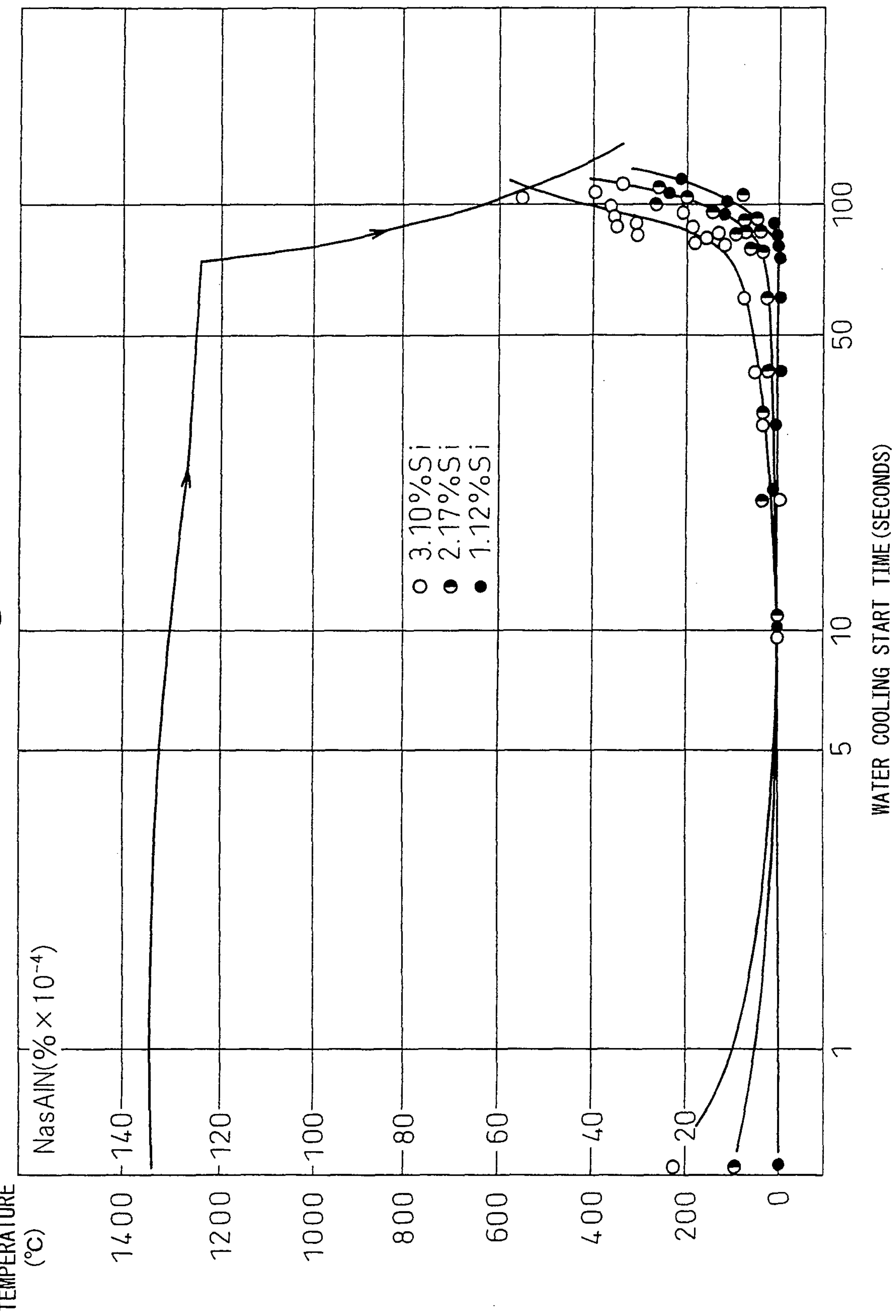

TEMPERATURE (°C)
B10 (T)
EXTRACTED BAR THICKNESS 40mm
3.10%Si
1300
1200
1100
1000
900
800
700
600
500
400
2.00
1.95
1.90
1.85
WATER COOLING
FINAL SHEET THICKNESS 3mm
d
c
e
b
a
WATER COOLING
WATER COOLING
AFTER HOT ROLLING
GRADUAL COOLING IN ATMOSPHERE
WATER COOLING
50
100
150
TIME UNTIL START OF RAPID COOLING AFTER END OF HOT ROLLING (SECONDS)

Fig.6


TEMPERATURE (°C)
1400
1200
1000
800
600
400
200
0
NasAlN(%×10⁻⁴)
140
120
100
80
60
40
20
1
5
10
50
100
WATER COOLING START TIME (SECONDS)
○ 3.10%Si
◐ 2.17%Si
● 1.12%Si

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2007/062183

A. CLASSIFICATION OF SUBJECT MATTER
B21B3/02(2006.01)i, *C21D8/12*(2006.01)i, *C21D9/46*(2006.01)i, *C22C38/00*(2006.01)i, *C22C38/60*(2006.01)i, *H01F1/16*(2006.01)i, *H01F41/02*(2006.01)i

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)
B21B1/00-3/02, C21D8/12, C21D9/46, C22C38/00, C22C38/00-38/60, H01F1/16, H01F41/02

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

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2007
Kokai Jitsuyo Shinan Koho	1971-2007	Toroku Jitsuyo Shinan Koho	1994-2007

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2-121704 A (NKK Corp.), 09 May, 1990 (09.05.90), (Family: none)	1-4

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

* Special categories of cited documents:
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"P" document published prior to the international filing date but later than the priority date claimed
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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
07 September, 2007 (07.09.07)	18 September, 2007 (18.09.07)

Name and mailing address of the ISA/	Authorized officer
Japanese Patent Office	
Facsimile No.	Telephone No.

Form PCT/ISA/210 (second sheet) (April 2005)

REFERENCES CITED IN THE DESCRIPTION

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- **Saito et al.** *Journal of the Japan Institute of Metals,* 1963, vol. 27, 191 **[0002]**
- **C. G. Dunn.** *Acta Metallurgica,* 1953, vol. 1, 163 **[0002]**
- *Ferrum,* 2004, vol. 9 (2), 52 **[0003]**
- *Material Science Forum,* 1996, vol. 204-206 (Pt1), 143 **[0006]**