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(54) **Method of making high silicon, low carbon regular grain oriented silicon steel**

Verfahren zum Herstellen von normal kornorientiertem Stahl mit hohem Silizium- und niedrigem Kohlenstoffgehalt

Procédé de fabrication d'acier ordinaire à haute teneur en silicium, à basse teneur en carbone et à grains orientés

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(56) References cited:
EP-A- 0 047 129 **EP-A- 0 253 904**
EP-A- 0 334 223 **FR-A- 2 228 855**
FR-A- 2 439 238

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Description**TECHNICAL FIELD**

The invention relates to a process for producing high silicon regular grain oriented electrical steel with low melt carbon and in thicknesses ranging from 14 mils (0.35 mm) to 6 mils (0.15 mm) or less, and more particularly to such a process including an intermediate anneal following the first cold rolling stage having a very short soak time and a two-part temperature-controlled cooling cycle, and preferably an ultra-rapid anneal prior to decarburization.

BACKGROUND ART

The teachings of the present invention are applied to silicon steel having a cube-on-edge orientation, designated (110) [001] by Miller's Indices. Such silicon steels are generally referred to as grain oriented electrical steels. Grain oriented electrical steels are divided into two basic categories: regular grain oriented and high permeability grain oriented. Regular grain oriented electrical steel utilizes manganese and sulfur (and/or selenium) as the principle grain growth inhibitor and generally has a permeability at 796 A/m of less than 1870. High permeability electrical steel relies on aluminum nitrides, boron nitrides or other species known in the art made in addition to or in place of manganese sulphides and/or selenides as grain growth inhibitors and has a permeability greater than 1870. The teachings of the present invention are applicable to regular grain oriented silicon steel.

Conventional processing of regular grain oriented electrical steel comprises the steps of preparing a melt of electrical steel in conventional facilities, refining and casting the electrical steel in the form of ingots or strand cast slabs. The cast electrical steel preferably contains in weight percent less than about 0.1% carbon, 0.025% to 0.25% manganese, 0.01% to 0.035% sulfur and/or selenium, 2.5% to 4.0% silicon with an aim silicon content of about 3.15%, less than 50 ppm nitrogen and less than 100 ppm total aluminum, the balance being essentially iron. Additions of boron and/or copper can be made, if desired.

If cast into ingots, the steel is hot rolled into slabs or directly rolled from ingots to strip. If continuous cast, the slabs may be pre-rolled in accordance with U.S. Patent 4,718,951. If developed commercially, strip casting would also benefit from the process of the present invention. The slabs are hot rolled at about 2550° F (1400° C) to hot band thickness and are subjected to a hot band anneal of about 1850° F (1010° C) with a soak of about 30 seconds. The hot band is air cooled to ambient temperature. Thereafter, the material is cold rolled to intermediate gauge and subjected to an intermediate anneal at a temperature of about 1740° F (950° C) with a 30 second soak and is cooled as by air cooling to ambient temperature. Following the intermediate anneal, electrical steel is cold rolled to final gauge. The electrical steel at final gauge is subjected to a conventional decarburizing anneal which serves to recrystallize the steel, to reduce the carbon content to a non-aging level and to form a fayalite surface oxide. The decarburizing anneal is generally conducted at a temperature of from 1525° F to 1550° F (830° C to 845° C) in a wet hydrogen bearing atmosphere for a time sufficient to bring the carbon content down to about 0.003% or lower. Thereafter, the electrical steel is coated with an annealing separator such as magnesia and is final annealed at a temperature of about 2200° F (1200° C) for twenty-four hours. This final anneal brings about secondary recrystallization. A forsterite or "mill" glass coating is formed by reaction of the fayalite layer with the separator coating.

Representative processes for producing regular grain oriented (cube-on-edge) silicon steel are taught in U.S. Patent Nos. 4,202,711; 3,764,406; and 3,843,422.

In recent years, to lower the core loss of regular grain oriented products, attention has been turned to increasing the volume resistivity by raising the silicon content to suppress macro-eddy current losses. However, the expected improvement from higher silicon content has generally not been realized. A typical prior art approach has been to increase both silicon and carbon in particular ratios in an attempt to achieve improved magnetic quality. It has been found that raising carbon and silicon together will make the steel more prone to incipient grain boundary melting during the high temperature ingot/slab heating process and more brittle in subsequent processing after hot rolling. Particularly the handling and cold rolling characteristics of the higher silicon and carbon material are degraded. In the process of making regular grain oriented silicon steel, decarburization to 0.003% carbon or less is required to provide nonaging magnetic properties in the finished grain oriented electrical steel. However, higher silicon retards decarburization, making high silicon, high melt carbon materials more difficult to produce.

The present invention is based upon the discovery that in the production of regular grain oriented electrical steel the nature of the intermediate anneal following first stage of cold rolling, and its cooling cycle, have a marked effect on the magnetic quality of the final product. The volume fraction of austenite formed during the anneal, the austenite decomposition product and the carbide precipitate formed during cooling are all of significant importance. A cooling rate after the intermediate anneal which does not allow for austenite decomposition subsequent to the precipitation of fine iron carbide produces lower permeability, less stable secondary grain growth, and/or an enlarged secondary grain size. Added to this, higher silicon will raise the activity of carbon, increasing the carbide precipitation temperature and

producing a coarser carbide. As a result, the problems created by improper cooling after the intermediate anneal are aggravated at higher silicon. The teachings of the present invention overcome these problems.

The present invention is directed to the production of regular grain oriented silicon steel starting with a melt chemistry having a silicon content of from 3% to 4.5% and a low carbon content of less than 0.07%. The routing of The present invention follows the conventional routing given above with two exceptions.

First, the present invention contemplates a modified intermediate anneal procedure following the first stage of cold rolling. The modified intermediate anneal procedure preferably has a short soak at a lower temperature than the typical prior art intermediate anneal and includes a temperature controlled, two-stage cooling cycle, as will be fully described hereinafter.

The intermediate anneal cooling practice of the present invention provides for austenite decomposition in the first slow stage of cooling prior to precipitation of fine iron carbide in the second rapid stage of cooling. The short soak feature and austenite decomposition are facilitated by the low melt carbon.

Second, the routing of the present invention preferably includes an ultra-rapid annealing treatment prior to decarburization. The ultra-rapid annealing treatment improves the overall magnetic quality by improving the recrystallization texture. The ultra-rapid annealing treatment is of the type set forth in U.S. Patent 4,898,626.

Briefly, U.S. Patent 4,898,626 teaches that the ultra-rapid annealing treatment is performed by heating the electrical steel at a rate in excess of 180° F (100° C) per second to a temperature above the recrystallization temperature, nominally 1250° F (675° C). The ultra-rapid annealing treatment can be performed at any point in the routing after at least a first stage of cold rolling and before the decarburization anneal preceding the final anneal. A preferred point in the routing is after the completion of cold rolling and before the decarburization anneal. The ultra-rapid annealing treatment may be accomplished either prior to the decarburization anneal, or may be incorporated into the decarburization anneal as a heat-up portion thereof.

DISCLOSURE OF THE INVENTION

According to the invention there is provided a method for processing regular grain oriented silicon steel having a thickness in the range of from 14 mils (0.35 mm) to 6 mils (0.15 mm) or less comprising the steps of providing electrical steel consisting essentially of, in weight percent, less than 0.07% carbon, 0.025% to 0.25% manganese, 0.01% to 0.035% sulfur and/or selenium, 3.0% to 4.5% silicon, less than 100 ppm total aluminum, less than 50 ppm nitrogen, the balance being essentially iron. Additions of boron and/or copper can be made, if desired.

To this end, the starting material referred to as "hot band" can be produced by a number of methods known in the art such as ingot casting/continuous casting and hot rolling, or by strip casting.

The hot band is subjected to an anneal at about 1850° F (1010° C) for a soak time of about 30 seconds, followed by air cooling to ambient temperature. It has been found that this hot band anneal can be omitted, particularly when making a regular grain oriented electrical steel having a silicon content at the lower portion of the range.

Thereafter, the electrical steel is cold rolled to intermediate gauge. The cold rolled intermediate thickness electrical steel is subjected to an intermediate anneal at 1650° F to 2100° F (900° C to 1150° C) and preferably from 1650° F to 1700° F (from 900° C to 930° C) for a soak time of from 1 to 30 seconds, and preferably from 3 to 8 seconds. Following this soak, the electrical steel is cooled in two stages. The first is a slow cooling stage from soak temperature to a temperature of from 1000° F to 1200° F (540° C to 650° C), and preferably to a temperature of 1100° F $\pm$ 50° F (595° C $\pm$ 30° C) at a rate less than 1500° F (835° C) per minute, and preferably at a rate of from 500° F (280° C) to 1050° F (585° C) per minute. The second stage is a fast cooling stage at a rate of greater than 1500° F (835° C) per minute and preferably at a rate of 2500° F to 3500° F (1390° C to 1945° C) per minute, followed by a water quench at 600° F to 1000° F (315° C to 540° C). Following the intermediate anneal, the electrical steel is cold rolled to final gauge, decarburized, coated with an annealing separator, and subjected to a final anneal to effect secondary recrystallization.

In a preferred practice of the invention, the electrical steel is subjected to an ultra-rapid annealing treatment of the type described above. This can be performed at any point in the routing after at least a first stage of cold rolling, and before decarburization. It is generally preferred to perform the ultra-rapid annealing treatment upon completion of cold rolling and before the decarburization anneal. As indicated above, the ultra-rapid anneal may be incorporated into the decarburization annealing step as a heat-up portion thereof.

BRIEF DESCRIPTION OF THE DRAWING

The Figure is a graph illustrating the intermediate anneal time/temperature cycle of the present invention and of a typical prior art intermediate anneal.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the practice of the present invention, the routing for the high silicon, low melt carbon regular grain oriented electrical steel is conventional and is essentially the same as that given above with three exceptions. The first exception is that the hot band anneal can be omitted, if desired. Where equipment and conditions permit, the practice of a hot band anneal is recommended since it makes the high silicon regular grain oriented electrical steel less brittle and more amenable to cold rolling. Furthermore, it tends to contribute to more stable secondary recrystallization. When practiced, a hot band anneal is provided at a temperature of about 1850° F (1010° C) at a soak time of about 30 seconds. The hot band anneal is followed by air cooling to ambient temperature. The second exception is the development of the intermediate anneal and cooling practice of the present invention following the first stage of cold rolling. Finally, the third exception is the optional, but preferred, use of an ultra-rapid annealing treatment prior to decarburization.

Following the first stage of cold rolling, the silicon steel is subjected to an intermediate anneal in accordance with the teachings of the present invention. Reference is made to the Figure, which is a schematic of the time/temperature cycle for the intermediate anneal of the present invention. The Figure also shows, with a broken line, the time/temperature cycle for a typical, prior art intermediate anneal.

A primary thrust of the present invention is the discovery that the intermediate anneal and its cooling cycle can be adjusted to provide a fine carbide dispersion. The anneal and its cooling cycle overcome the adverse effects of a higher silicon content, described above.

During the heat-up portion of the intermediate anneal, recrystallization occurs at about 1250° F (675° C), roughly 20 seconds after entering the furnace, after which normal grain growth occurs. The start of recrystallization is indicated at "O" in the Figure. Above about 1280° F (690° C) carbides will begin dissolving, as indicated at "A" in the Figure. This event continues and accelerates as the temperature increases. Above about 1650° F (900° C), a small amount of ferrite transforms to austenite. The austenite provides for more rapid solution of carbon and restricts normal grain growth, thereby establishing the intermediate annealed grain size. Prior art intermediate anneal practice provided a soak at about 1740° F (950° C) for a period of at least 25 to 30 seconds. The intermediate anneal procedure of the present invention provides a soak time of from 1 to 30 seconds, and preferably from 3 to 8 seconds. The soak temperature has been determined not to be critical. The soak can be conducted at a temperature of from 1650° F (900° C) to 2100° F (1150° C). Preferably, the soak is conducted at a temperature of from 1650° F (900° C) to 1700° F (930° C), and more preferably at about 1680° F (915° C). The shorter soak time and the lower soak temperature are preferred because less austenite is formed. Further, the austenite present in the form of dispersed islands at the prior ferrite grain boundaries is finer. Thus, the austenite is easier to decompose into ferrite with carbon in solid solution for subsequent precipitation of fine iron carbide. To extend either the soak temperature or time results in the enlargement of the austenite islands which rapidly become carbon-rich compared to the prior ferrite matrix. Both growth and carbon enrichment of the austenite hinder its decomposition during cooling. The desired structure exiting the furnace consists of a recrystallized matrix of ferrite having less than about 5% austenite uniformly dispersed throughout the material as fine islands. At the end of the anneal, the carbon will be in solid solution and ready for reprecipitation on cooling. The primary reason behind the redesign of the intermediate anneal time and temperature at soak is the control of the growth of the austenite islands. The lower temperature reduces the equilibrium volume fraction of austenite which forms. The shorter time reduces carbon diffusion, thereby inhibiting growth and undue enrichment of the austenite. The lower strip temperature, the reduced volume fraction and the finer morphology of the austenite make it easier to decompose during the cooling cycle.

Immediately after the soak, the cooling cycle is initiated. The cooling cycle of the present invention contemplates two stages. The first stage extending from soak to the point "E" on the Figure is a slow cool from soak temperature to a temperature of from 1000° F (540° C) to 1200° F (650° C) and preferably to 1100° ± 50° F (595° C ± 30° C). This first slow cooling stage provides for the decomposition of austenite to carbon-saturated ferrite. Under equilibrium conditions, austenite decomposes to carbon-saturated ferrite between from about 1650° F (900° C) and 1420° F (770° C). However, the kinetics of the cooling process are such that austenite decomposition does not begin in earnest until the mid 1500° F (815° C) range and continues somewhat below 1100° F (595° C).

Failure to decompose the austenite in the first cooling stage will result in the formation of martensite and/or pearlite. Martensite, if present, will cause an enlargement of the secondary grain size, and the deterioration of the quality of the (110)[001] orientation. Its presence adversely affects energy storage in the second stage of cold rolling, and results in poorer and more variable magnetic quality of the final electrical steel product. Lastly, martensite degrades the mechanical properties, particularly the cold rolling characteristics. Pearlite is more benign, but still ties up carbon in an undesired form.

As indicated above, austenite decomposition begins at about point "C" in the Figure and continues to about point "E". At point "D" fine iron carbide begins to precipitate from the carbon-saturated ferrite. Under equilibrium conditions, carbides begin to precipitate from carbon-saturated ferrite at temperatures below 1280° F (690° C). However, the actual process requires some undercooling to start precipitation, which begins in earnest at about 1200° F (650° C). It will be

noted that the austenite decomposition to carbon-rich ferrite and carbide precipitation from the ferrite overlap somewhat. The carbide is in two forms. It is present as an intergranular film and as a fine intragranular precipitate. The former precipitates at temperatures above about 1060° F (570° C). The latter precipitates below about 1060° F (570° C). The slow cooling first stage, extending from point "C" to point "E" of the Figure has a cooling rate of less than 1500° F (835° C) per minute, and preferably from 500° F to 1050° F (280° C to 585° C) per minute.

The second stage of the cooling cycle, a fast cooling stage, begins at point "E" in the Figure and extends to point "G" between 600° F and 1000° F (315° C and 540° C) at which point the strip can be water quenched to complete the rapid cooling stage. The strip temperature after water quenching is 150° F (65° C) or less, which is shown in the Figure as room temperature (75° F or 25° C). During the second cooling stage, the cooling rate is preferably from 2500° F to 3500° F (1390° C to 1945° C) per minute and preferably greater than 3000° F (1665° C) per minute. This assures the precipitation of fine iron carbide.

It will be evident from the above that the entire intermediate anneal and cooling cycle of the present invention is required in the process of obtaining the desired microstructure, and precise controls are critical. The typical prior art cycle time shown in the Figure required at least 3 minutes, terminating in a water bath, not shown, at a strip speed of about 220 feet per minute (57 meters per minute). The intermediate anneal cycle time of the present invention requires about 2 minutes, 10 seconds which enabled a strip speed of about 260 feet per minute (80 meters per minute) to be used. It will therefore be noted that the annealing cycle of the present invention enables greater productivity of the line. No aging treatment after the anneal is either needed or desired, since it has been found to cause the formation of an enlarged secondary grain size which degrades the magnetic quality of the final electrical steel product.

The intermediate anneal is followed by the second stage of cold rolling reducing the electrical steel to the desired final gauge. At this stage, the electrical steel can be decarburized, coated with an annealing separator and subjected to a final anneal to effect secondary recrystallization.

In the preferred practice of the present invention, the electrical steel is given an ultra-rapid annealing treatment after cold reduction and prior to decarburization. To this end, the electrical steel at final gauge is heated at a rate above 180° F (100° C) per second to a temperature above 1250° F (675° C). Preferably, the electrical steel is heated at a rate of 1000° F (540° C) per second. It is additionally preferred that the ultra-rapid annealing treatment be performed as a heat-up portion of the decarburizing anneal.

The preferred chemistry of the present invention in weight % is as follows: less than 0.05% carbon, 0.04% to 0.08% manganese, 0.015% to 0.025% sulfur and/or selenium, 3.25% to 3.75% silicon, less than 100 ppm aluminum, less than 50 ppm nitrogen, additions of boron and/or copper, can be made if desired, the balance being essentially iron.

The ultra-rapid annealing treatment improves the recrystallization texture after decarburization by creating more (110)[001] primary grains. It also contributes to smaller secondary grain size. When an ultra-rapid annealing treatment is incorporated into the process, the process is less sensitive to intermediate and final gauge variations and the magnetic characteristics of the regular grain oriented silicon steel are improved and more consistent.

EXAMPLE I

Four heats were melted having the compositions in weight % shown in Table I. The heats were prepared by continuous casting into 8" (200 mm) thick slabs, prerolling the 8" thick slabs to 6" (150 mm), reheating to 2550° F (1400° C) and hot rolling to 0.084" (2.1 mm) hot bands for subsequent processing. The plant processing followed a routing using a 1850° F (1010° C) hot band annealing treatment and cold rolling to various intermediate thicknesses; however, Heats A and B were processed using a typical prior art intermediate anneal with a 1740° F (950° C) soak for 25-30 seconds followed by normal ambient cooling while Heats C and D were intermediate annealed according to the practice of the present invention. After intermediate annealing, the materials were cold rolled to final thicknesses of 7-mils (0.18 mm) and 9-mils (0.28 mm). After completing cold rolling, the materials were decarburized at 1525° F (830° C) in a wet hydrogen-bearing atmosphere, MgO coated and given a final anneal at 2200° F (1200° C). The resulting magnetic quality obtained in these trials are summarized in Table III.

TABLE I

Code	C	Mn	S	Si	Al	Cu	P	N
A	0.0288	0.059	0.0198	3.41	0.0013	0.092	0.006	0.0042
B	0.0296	0.059	0.0209	3.42	0.0014	0.118	0.006	0.0038
C	0.0265	0.058	0.0218	3.44	0.0012	0.097	0.005	0.0040
D	0.0274	0.058	0.0212	3.36	0.0012	0.085	0.006	0.0035

TABLE II

	Heat	Hot Band End	Inter-mediate Thickness	P15	H-10	Inter-mediate Thickness	P15	H-10
Conventional Practice:	A	Front	0.020"	0.393	1842	0.022"	0.413	1849
		Back	"	0.396	1833	"	0.442	1831
	B	Front	"	0.399	1842	"	0.432	1842
		Back	"	0.420	1824	"	0.430	1840
Present Invention with Conventional Decarburization:	C	Front	0.019"	0.383	0844	0.021"	0.411	1845
		Back	"	0.380	1838	"	0.412	1843
	D	Front	"	0.376	1845	"	0.408	1844
		Back	"	0.381	1840	"	0.410	1840
	C	Front	0.021"	0.373	1841	0.023"	0.411	1846
		Back	"	0.380	1838	"	0.423	1836
	D	Front	"	0.368	1849	"	0.402	1849
		Back	"	0.379	1840	"	0.405	1846
Averages:	C	Front	0.025"	0.376	1838	0.025"	0.405	1844
		Back	"	0.376	1840	"	0.407	1846
	D	Front	"	0.377	1841	"	0.405	1846
		Back	"	0.376	1837	"	0.406	1845
Improvement of Present Invention:	Conventional Practice		0.022"	0.402	1835		0.429	1841
	Present Invention:		0.019"	0.380	1842		0.410	1843
	"		0.021"	0.375	1842		0.410	1844
	"		0.025"	0.376	1839		0.406	1845
			5.5%					
			6.7%					
			6.4%					

The results clearly show that the practice of the intermediate anneal cycle of the present invention provided improved core loss and enhanced stability of secondary grain growth for these regular grain oriented materials.

EXAMPLE II

Additional samples from Heats A and B were secured during plant processing trials for laboratory processing. Plant processing followed the conventional routing of example I; however, after cold rolling to intermediate thickness was completed, the samples were secured in the plant and processed in the laboratory in accordance with the teachings of the present invention wherein the intermediate annealing soak temperatures and times and controlled cooling prac-

tice were employed and the more preferred practice utilizing an ultra-rapid annealing treatment after completion of cold rolling and prior to decarburization was employed. In the practice of the latter, a 1000° F (556° C) per second heating rate from room temperature to 1375° F was incorporated into the heat-up portion of the decarburization anneal. After the intermediate anneal, the materials were cold rolled to 7-mils (0.18 mm) final thickness and decarburized at 1525° F (830° C) in a wet hydrogen-bearing atmosphere using either conventional techniques and ultra-rapid annealing treatment during heating. After decarburization, the samples were MgO coated and given a final anneal at 2200° F (1200° C). The results of these runs are summarized in Table III.

TABLE III

Heat	Hot Band End	Inter-mediate Thickness	P15	H10
Conventional Practice:				
A	Front	0.020"	0.395	1847
	Back	"	0.391	1837
B	Front	"	0.399	1842
	Back	"	0.420	1824
Present Invention w/Conventional Decarburization:				
A	Front	0.021"	0.368	1846
	Back	to 0.024"	0.359	1850
B	Front	0.024"	0.372	1855
	Back		0.363	1855
Present Invention w/Ultra-Rapid Annealing:				
A	Front	0.021"	0.355	1853
	Back	to 0.024"	0.350	1856
B	Front	0.024"	0.359	1859
	Back		0.353	1857
Conventional practice				
Pres. Invention - Conventional Decarburization			0.401	1838
Pres. Invention - Ultra-Rapid Annealing			0.366	1857
			0.354	1856

Improvement of Present Invention:

The results clearly show that the practice of the intermediate anneal cycle of the present invention provided improved core loss and enhanced the stability of secondary grain growth for these regular grain oriented materials. The more preferred practice whereby an ultra-rapid annealing treatment in addition to the intermediate anneal cycle of the present invention further provided for still more improvement in the magnetic quality.

Claims

1. A process for producing high silicon, low melt carbon, regular grain oriented electrical steel having a thickness of from 14 mils (0.35 mm) to 6 mils (0.15 mm) or less, comprising the steps of providing a hot band of silicon steel containing in weight percent from 3.0% to 4.5% silicon and less than 0.07% carbon, annealing said hot band, removing the hot band scale if required, cold rolling to intermediate gauge, subjecting said intermediate gauge

material to an intermediate anneal at a soak temperature of from 1650° F (900° C) to 2100° F (1150° C) for a soak time of from 1 second to 30 seconds, conducting a slow cooling stage from said soak temperature to a temperature of from 1000° F (540° C) to 1200° F (650° C) at a cooling rate less than 1500° F (835° C) per minute, thereafter conducting a fast cooling stage to a temperature of from 600° F (315° C) to 1000° F (540° C) at a rate greater than 1500° F (835° C) per minute followed by a water quench, cold rolling said silicon steel to final gauge, subjecting said final gauge silicon steel to a decarburizing anneal, coating said decarburized silicon steel with an annealing separator, and subjecting said silicon steel to a final anneal to effect secondary recrystallization.

2. The process claimed in claim 1 wherein said silicon content in weight percent is 3.25%-3.75%.
3. The process claimed in claim 1 wherein said hot band anneal is conducted at a temperature of about 1850° F (1010° C) with a soak time of about 30 seconds and air cooling to ambient temperature.
4. The process in claim 1 including the step of subjecting said silicon steel at a final gauge and before decarburization to an ultra-rapid annealing treatment to a temperature greater than 1250° F (675° C) at a heating rate greater than 180° F (100° C) per second.
5. The process claimed in claim 1 including the step of conducting said intermediate anneal with a soak time of from 3 to 8 seconds.
6. The process claimed in claim 1 including the step of conducting said intermediate anneal at a soak temperature of from 1650° F (900° C) to 1700° F (930° C).
7. The process claimed in claim 1 including the step of conducting said intermediate anneal at a soak temperature of about 1680° F (915° C).
8. The process claimed in claim 1 including the step of terminating said slow cooling stage at a temperature of 1100° ± 50° F (595° C ± 30° C).
9. The process claimed in claim 1 including the step of conducting said slow cooling stage at a cooling rate of from 500° F (280° C) to 1050° F (585° C) per minute.
10. The process claimed in claim 1 including the step of conducting said fast cooling stage at a cooling rate of 2500° F (1390° C) to 3500° F (1945° C) per minute.
11. The process claimed in claim 1 including the steps of conducting said intermediate anneal with a soak temperature of about 1680° F (915° C) for a soak time of 3 to 8 seconds, conducting said slow cooling stage at a cooling rate of 500° F (280° C) to 1050° F (585° C) per minute, terminating said slow cooling stage at a temperature of 1100° F ± 50° F (595° C ± 30° C), and conducting said fast cooling stage at a rate of from 2500° F (1390° C) to 3500° F (1945° C) per minute.
12. The process claimed in claim 1 wherein said silicon steel consists essentially of, in weight percent, less than 0.07% carbon, 0.025% to 0.25% manganese, 0.01% to 0.035% sulfur and/or selenium, 3.0% to 4.5% silicon, less than 100 ppm aluminum, less than 50 ppm nitrogen, additions of boron and/or copper, if desired, the balance being essentially iron.
13. The process claimed in claim 1 including the step of subjecting said silicon steel at final gauge and before decarburization to an ultra-rapid annealing treatment to a temperature greater than 1250° F (675° C) at a heating rate greater than 180° F (100° C) per second.
14. The process claimed in claim 11 wherein said hot band anneal is conducted at a temperature of about 1850° F (1010° C) with a soak of about 30 seconds and air cooling to ambient temperature.
15. The process claimed in claim 13 including the step of performing said ultra-rapid annealing treatment as a heat-up portion of said decarburizing anneal.
16. The process claimed in claim 14 including the step of subjecting said silicon steel at final gauge and before decarburization to an ultra-rapid annealing treatment to a temperature greater than 1250° F (675° C) at a heating

rate greater than 180° F (100° C) per second.

17. The process claimed in claim 16 including the step of performing said ultra-rapid annealing treatment as a heat-up portion of said decarburizing anneal.

18. The process claimed in claim 1 wherein said silicon steel consists essentially of, in weight percent, less than 0.05% carbon, 0.04% to 0.08% manganese, 0.015% to 0.025% sulfur and/or selenium, 3.25% to 3.75% silicon, less than 100 ppm aluminum, less than 50 ppm nitrogen, additions of boron and/or copper, if desired, the balance being essentially iron.

Patentansprüche

1. Verfahren zur Herstellung von regulär-kornorientiertem Elektrostahl mit einem hohen Anteil an Silizium und einem niedrigen Anteil an geschmolzenem Kohlenstoff sowie einer Stärke von 14 mil (0,35 mm) bis 6 mil (0,15 mm) oder weniger, das folgende Schritte umfaßt: Bereitstellung eines Warmwalzbandes aus Siliziumstahl mit 3,0 bis 4,5 Gew.-% Silizium und weniger als 0,07 Gew.-% Kohlenstoff, Glühen des Warmwalzbandes, gegebenenfalls Entfernung des Walzzunders vom Warmwalzband, Kaltwalzen auf eine mittlere Stärke, Zwischenglühen dieses Materials mittlerer Stärke bei einer Durchwärmtemperatur von 1650° F (900° C) bis 2100° F (1150° C) und einer Durchwärmzeit von 1 Sekunde bis 30 Sekunden, langsames Abkühlen des Materials von der Durchwärmtemperatur auf eine Temperatur von 1000° F (540° C) bis 1200° F (650° C) mit einer Abkühlgeschwindigkeit von weniger als 1500° F (835° C) pro Minute, danach schnelles Abkühlen auf eine Temperatur von 600° F (315° C) bis 1000° F (540° C) mit einer Abkühlgeschwindigkeit von mehr als 1500° F (835° C) pro Minute und anschließendes Abschrecken mit Wasser, Kaltwalzen des Siliziumstahls auf die endgültige Stärke, Entkohlen des Siliziumstahls endgültiger Stärke, Überziehen des entkohlten Siliziumstahls mit einem Glühtrennmittel und endgültiges Glühen des Siliziumstahls zum Zwecke der Nachkristallisation.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Siliziumgehalt 3,25-3,75 Gew.-% beträgt.

3. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Warmwalzband bei einer Temperatur von etwa 1850° F (1010° C) während einer Durchwärmzeit von etwa 30 Sekunden geglüht und mit Luft auf Raumtemperatur abgekühlt wird.

4. Verfahren nach Anspruch 1, ferner gekennzeichnet durch Ultraschallglühen des Siliziumstahls endgültiger Stärke vor dem Entkohlen mit einer Erwärmungsgeschwindigkeit von mehr als 180° F (100° C) pro Sekunde auf eine Temperatur von mehr als 1250° F (675° C).

5. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Zwischenglühen während einer Durchwärmzeit von 3 bis 8 Sekunden erfolgt.

6. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Zwischenglühen bei einer Durchwärmtemperatur von 1650° F (900° C) bis 1700° F (930° C) erfolgt.

7. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Zwischenglühen bei einer Durchwärmtemperatur von etwa 1680° F (915° C) erfolgt.

8. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die langsame Abkühlung bei einer Temperatur von 1100° F $\pm$ 50° F (595° C $\pm$ 30° C) beendet wird.

9. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die langsame Abkühlung mit einer Abkühlgeschwindigkeit von 500° F (280° C) bis 1050° F (585° C) pro Minute erfolgt.

10. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die schnelle Abkühlung mit einer Abkühlgeschwindigkeit von 2500° F (1390° C) bis 3500° F (1945° C) pro Minute erfolgt.

11. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Zwischenglühen bei einer Durchwärmtemperatur von etwa 1680° F (915° C) während einer Durchwärmzeit von 3 bis 8 Sekunden erfolgt, das langsame Abkühlen mit einer Abkühlgeschwindigkeit von 500° F (280° C) bis 1050° F (585° C) pro Minute erfolgt und bei einer Tem-

peratur von 1100° F ± 50° F (595° C ± 30° C) beendet wird, und daß die schnelle Abkühlung mit einer Abkühlgeschwindigkeit von 2500° F (1390° C) bis 3500° F (1945° C) pro Minute erfolgt.

12. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Siliziumstahl im wesentlichen aus weniger als 0,07 Gew.-% Kohlenstoff, 0,025 bis 0,25 Gew.-% Mangan, 0,01 bis 0,035 Gew.-% Schwefel und/oder Selen, 3,0 bis 4,5 Gew.-% Silizium, weniger als 100 ppm Aluminium, weniger als 50 ppm Stickstoff, gegebenenfalls Bor- und/oder Kupferzusätzen, und zum Rest im wesentlichen aus Eisen besteht.
13. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Siliziumstahl endgültiger Stärke vor dem Entkohlen in einem Ultraschnellglühverfahren mit einer Erwärmungsgeschwindigkeit von mehr als 180° F (100° C) pro Sekunde auf eine Temperatur von mehr als 1250° F (675° C) gebracht wird.
14. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß das Warmwalzband bei einer Temperatur von etwa 1850° F (1010° C) während einer Durchwärmzeit von etwa 30 Sekunden geglüht und mit Luft auf Raumtemperatur abgekühlt wird.
15. Verfahren nach Anspruch 13, dadurch gekennzeichnet, daß das Ultraschnellglühen als eine Erwärmungsphase der Entkohlung durchgeführt wird.
16. Verfahren nach Anspruch 14, dadurch gekennzeichnet, daß der Siliziumstahl endgültiger Stärke vor dem Entkohlen in einem Ultraschnellglühverfahren mit einer Erwärmungsgeschwindigkeit von mehr als 180° F (100° C) pro Sekunde auf eine Temperatur von mehr als 1250° F (675° C) gebracht wird.
17. Verfahren nach Anspruch 16, dadurch gekennzeichnet, daß das Ultraschnellglühen als eine Erwärmungsphase der Entkohlung durchgeführt wird.
18. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Siliziumstahl im wesentlichen aus weniger als 0,05 Gew.-% Kohlenstoff, 0,04 bis 0,08 Gew.-% Mangan, 0,015 bis 0,025 Gew.-% Schwefel und/oder Selen, 3,25 bis 3,75 Gew.-% Silizium, weniger als 100 ppm Aluminium, weniger als 50 ppm Stickstoff, gegebenenfalls Bor- und/oder Kupferzusätzen, und zum Rest im wesentlichen aus Eisen besteht.

Revendications

1. Procédé de production d'acier électrique ordinaire à haute teneur en silicium, à faible teneur en carbone fondu et à grains orientés ayant une épaisseur de 0,35 mm (14 mils) à 0,15 mm (6 mils) ou moins, comprenant les étapes consistant à mettre en oeuvre une bande chaude d'acier au silicium contenant 3,0 à 4,5% en poids de silicium et moins de 0,07% en poids de carbone, à recuire ladite bande chaude, à éliminer la calamine de la bande chaude si c'est nécessaire, à laminier à froid à un calibre intermédiaire, à soumettre ledit matériau à calibre intermédiaire à un recuit intermédiaire à une température de trempe de 900°C (1650°F) à 1150°C (2100°F) pendant une période de trempe de 1 à 30 secondes, à réaliser une étape de refroidissement lente à partir de ladite température de trempe jusqu'à une température de 540°C (1000°F) à 650°C (1200°F) à une cadence de refroidissement inférieure à 835°C (1500°F) par minute, à réaliser ensuite une étape de refroidissement rapide jusqu'à une température de 315°C (600°F) à 540°C (1000°F) à une cadence supérieure à 835°C (1500°F) par minute. le tout étant suivi d'une trempe à l'eau, à laminier à froid ledit acier au silicium jusqu'au calibre final, à soumettre ledit acier de silicium au calibre final à un recuit de décarburation, à revêtir ledit acier au silicium décarburé par un séparateur de recuit et à soumettre ledit acier de silicium à un recuit final pour effectuer une recristallisation secondaire.
2. Procédé selon la revendication 1, dans lequel ladite teneur en silicium est de 3,25 à 3,75% en poids.
3. Procédé selon la revendication 1, dans lequel ledit recuit de la bande chaude est réalisé à une température d'environ 1010°C (1850°F) avec une durée de trempe d'environ 30 secondes et un refroidissement à l'air jusqu'à la température ambiante.
4. Procédé selon la revendication 1, comprenant l'étape consistant à soumettre ledit acier au silicium, au calibre final et avant décarburation, à un traitement de recuit ultra-rapide jusqu'à une température supérieure à 675°C (1250°F) à une cadence de chauffage supérieure à 100°C (180°F) par seconde.

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5. Procédé selon la revendication 1, comprenant l'étape consistant à réaliser ledit recuit intermédiaire avec une durée de trempe de 3 à 8 secondes.
- 5 6. Procédé selon la revendication 1, comprenant l'étape consistant à réaliser ledit recuit intermédiaire à une température de trempe de 900°C (1650°F) à 930°C (1700°F).
7. Procédé selon la revendication 1, comprenant l'étape consistant à réaliser ledit recuit intermédiaire à une température de trempe d'environ 915°C (1680°F).
- 10 8. Procédé selon la revendication 1, comprenant l'étape consistant à terminer ladite étape de refroidissement lente à une température de 595°C plus ou moins 30°C (1100°F plus ou moins 50°F).
9. Procédé selon la revendication 1, comprenant l'étape consistant à effectuer ladite étape de refroidissement lente à une cadence de refroidissement de 280°C (500°F) à 585°C (1050°F) par minute.
- 15 10. Procédé selon la revendication 1, comprenant l'étape consistant à réaliser ladite étape de refroidissement rapide à une cadence de refroidissement de 1390°C (2500°F) à 1945°C (3500°F) par minute.
- 20 11. Procédé selon la revendication 1, comprenant les étapes consistant à effectuer ledit recuit intermédiaire à une température de trempe d'environ 915°C (1680°F) pendant une période de trempe de 3 à 8 secondes. à réaliser ladite étape de refroidissement lente à une cadence de refroidissement de 280°C (500°F) à 585°C (1050°F) à la minute, à terminer ladite étape de refroidissement lente à une température de 595°C plus ou moins 30°C (1100°F plus ou moins 50°F) et à réaliser ladite étape de refroidissement rapide à une cadence de 1390°C (2500°F) à 1945°C (3500°F) par minute.
- 25 12. Procédé selon la revendication 1, dans lequel ledit acier au silicium est constitué essentiellement, en pourcentage en poids, de moins de 0.07% de carbone, de 0.025 à 0.25% de manganèse, de 0,01 à 0,035% de soufre et/ou de sélénium, de 3.0 à 4.5% de silicium, de moins de 100 ppm d'aluminium, de moins de 50 ppm d'azote, d'additions de bore et/ou de cuivre, si on le souhaite, le restant étant essentiellement du fer.
- 30 13. Procédé selon la revendication 1, comprenant l'étape consistant à soumettre ledit acier au silicium, au calibre final et avant décarburation, à un traitement de recuit ultra-rapide jusqu'à une température supérieure à 675°C (1250°F) à une cadence de chauffage supérieure à 100°C (180°F) par seconde.
- 35 14. Procédé selon la revendication 11, dans lequel ledit recuit de la bande chaude est réalisé à une température d'environ 1010°C (1850°F) avec une période de trempe d'environ 30 secondes et un refroidissement à l'air jusqu'à la température ambiante.
- 40 15. Procédé selon la revendication 13, comprenant l'étape consistant à effectuer ledit traitement de recuit ultra-rapide en tant que partie de réchauffement dudit recuit de décarburation.
- 45 16. Procédé selon la revendication 14, comprenant l'étape consistant à soumettre ledit acier au silicium, au calibre final et avant décarburation, à un traitement de recuit ultra-rapide jusqu'à une température supérieure à 675°C (1250°F) à une cadence de chauffage supérieure à 100°C (180°F) par seconde.
- 50 17. Procédé selon la revendication 16, comprenant l'étape consistant à effectuer ledit traitement de recuit ultra-rapide en tant que partie de réchauffement dudit recuit de décarburation.
- 55 18. Procédé selon la revendication 1, dans lequel ledit acier au silicium comprend essentiellement, en pourcentage en poids, moins de 0.05% de carbone, 0.04 à 0.08% de manganèse, 0,015 à 0,025% de soufre et/ou de séléniums, 3,25 à 3,75% de silicium, moins de 100 ppm d'aluminium, moins de 50 ppm d'azote, des additions de bore et/ou de cuivre, si on le souhaite, le restant étant essentiellement du fer.

