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[54] **PROCESS EMPLOYING COOLING IN A STATIC ATMOSPHERE FOR HIGH PERMEABILITY SILICON STEEL COMPRISING COPPER**

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[52] **U.S. Cl.** .... **148/112**; 75/123 L; 148/121

[51] **Int. Cl.**[2] **H01F 1/04**

[58] **Field of Search** .... 148/112, 111, 110, 120, 148/121, 31.55; 75/123 L

[56] **References Cited**

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[57] **ABSTRACT**

A process for producing silicon steel having a cube-on-edge orientation and a permeability of at least 1850 (G/O_e) at 10 oersteds, which includes the steps of: preparing a melt of steel consisting essentially of, by weight, up to 0.07% carbon, from 2.60 to 4.0% silicon, from 0.03 to 0.24% manganese, at least 0.01% selenium, from 0.01 to 0.09% of material from the group consisting of sulfur and selenium, from 0.015 to 0.04% aluminum, up to 0.02% nitrogen, from 0.1 to 0.5% copper, balance iron; casting the steel; hot rolling the steel; annealing the steel prior to a final cold roll at a temperature of from 1400° to 2150°F; cooling the steel from a temperature below 1700°F and above 750°F to a temperature at least as low as 500°F with a liquid quenching medium or gaseous stream and from its maximum annealing temperature to the temperature below 1700°F and above 750°F at a rate which is no faster than one wherein the steel is cooled in a static atmosphere or in a continuous processing line where there is some relative motion between the atmosphere and the steel, although the only deliberate motion is that imparted to the steel; and cold rolling the steel at a reduction of at least 80%.

15 Claims, No Drawings

PROCESS EMPLOYING COOLING IN A STATIC ATMOSPHERE FOR HIGH PERMEABILITY SILICON STEEL COMPRISING COPPER

The present invention relates to a process for producing electromagnetic silicon steel having a cube-on-edge orientation and a permeability of at least 1850 (G/O_e) at 10 oersteds.

Oriented silicon steels containing 2.60 to 4.0% silicon are generally produced by processes which involve hot rolling, a double cold reduction, an anneal before each cold roll and a high temperature texture anneal. Characterizing these steels are permeabilities at 10 oersteds of from about 1790 to 1840 (G/O_e).

In recent years a number of patents have disclosed methods for producing silicon steels with permeabilities in excess of 1850 (G/O_e) at 10 oersteds. Of these U.S. Pat. Nos. 3,287,183, 3,632,456 and 3,636,579 appear to be the most interesting. A still more interesting method is, however, described in a copending U.S. patent application. The application, Ser. No. 357,974, was filed on May 7, 1973 in the names of James A. Salsgiver and Frank A. Malagari. Application Ser. No. 357,974 describes a process which includes the steps of: preparing a melt of steel consisting essentially of, by weight, up to 0.07% carbon, from 2.6 to 4.0% silicon, from 0.03 to 0.24% manganese, from 0.01 to 0.07% sulfur, from 0.015 to 0.04% aluminum, up to 0.02% nitrogen, from 0.1 to 0.5% copper, balance iron; casting the steel; hot rolling the steel; annealing the steel prior to a final cold roll at a temperature of from 1400° to 2150°F; cooling the steel from a temperature below 1700°F and about 750°F to a temperature at least as low as 500°F with a liquid quenching medium or gaseous stream and from its maximum annealing temperature to the temperature below 1700°F and above 750°F at a rate which is no faster than one wherein the steel is cooled in a static atmosphere or in a continuous processing line where there is some relative motion between the atmosphere and the steel, although the only deliverate motion is that imparted to the steel; and cold rolling the steel at a reduction of at least 80%.

Described herein is another, and improved method for producing silicon steel having a cube-on-edge orientation and a permeability of at least 1850 (G/O_e) at 10 oersteds. It is primarily based upon the discovery that the melt of application Ser. No. 357,974 can be prepared with selenium replacing part or all of the sulfur contained therein.

It is accordingly an object of the present invention to provide a process for producing electromagnetic silicon steel having a cube-on-edge orientation and a permeability of at least 1850 (G/O_e) at 10 oersteds.

The present invention provides a method for producing silicon steel having a cube-on-edge orientation and a permeability of at least 1850 (G/O_e) at 10 oersteds. Involved therein are the steps of: preparing a melt of silicon steel consisting essentially of, by weight, up to 0.07% carbon, from 2.60 to 4.0% silicon, from 0.03 to 0.24% manganese, at least 0.01% selenium, from 0.01 to 0.09% of material from the group consisting of sulfur and selenium, from 0.015 to 0.04% aluminum, up to 0.02% nitrogen, from 0.1 to 0.5% copper, balance iron; casting the steel; hot rolling the steel into a hot rolled band; subjecting the steel to at least one cold rolling; subjecting the steel to final annealing prior to the final cold rolling; decarburizing the steel; and final texture annealing the steel. Aslo included, and significantly so, are the specific steps of: carrying out the final anneal prior to the final cold rolling at a temperature of from 1400° to 2150°F for a period of from 15 seconds to 2 hours; cooling the steel from a temperature below 1700°F and above 750°F to a temperature at least as low as 500°F with a liquid quenching medium or gaseous stream and from its maximum annealing temperature to the temperature below 1700°F and above 750°F at a rate which is no faster than one wherein the steel is cooled in a static atmosphere or in a continuous processing line where there is some relative motion between the atmosphere and the steel, although the only deliverate motion is that imparted to the steel; and cold rolling the cooled steel at a reduction of at least 80%. Preferred conditions include annealing at a temperature of from 1800° to 2125°F, cooling with a liquid quenching medium or gaseous stream from a temperature below 1600°F and above 1000°F, and cold rolling at a reduction of at least 85%.

Melting, casting, hot rolling, cold rolling, decarburizing and final texture annealing do not involve any novel procedure, as far as techniques are concerned, and with regard to them, the invention encompasses all applicable steelmaking procedures. As to the cold rolling, it should, however, be pointed out that several roll passes can constitute a single cold rolling operation, and that plural cold rolling operations exist only when cold rolling passes are separated by an anneal.

The steel melt must include silicon, aluminum, manganese and selenium. Silicon is necessary as it increases the steel's resistivity, decreases its magnetostriction, decreases its magnetocrystalline anisotropy and hence decreases its core loss. Aluminum, manganese and selenium are necessary as they form inhibitors which are essential for controlling the steel's orientation and its properties which are dependent thereon. More specifically, aluminum combines with nitrogen in the steel or from the atmosphere, to form aluminum nitride; and manganese combines with selenium, and possibly copper, to form manganese selenide and/or manganese copper selenide, and with sulfur if it is present, to form manganese sulfide and/or manganese copper sulfide. All together, these compounds inhibit normal grain growth during the final texture anneal, while at the same time aiding in the development of secondary recrystallized grains having the desired cube-on-edge orientation. Copper, noted above for its presence in manganese inhibitors, can also be beneficial during processing. It is hypothesized that copper can lower the annealing temperature, lower the temperature from which the rapid cool can occur, improve rollability, simplify melting, and relax annealing atmosphere requirements. Moreover, copper increases the steel's resistivity and decreases its core loss.

A steel in which the process of the present invention is particularly adaptable to consists essentially of, by weight, from 0.02 to 0.07% carbon, from 2.65 to 3.25% silicon, from 0.05 to 0.20% manganese, at least 0.02% selenium, from 0.02 to 0.07% of material from the group consisting of sulfur and selenium, from 0.015 to 0.04% aluminum, from 0.0030 to 0.0090% nitrogen, from 0.1 to 0.4% copper, balance iron. This steel has its chemistry balanced so as to produce a highly beneficial structure when processed according to the present invention.

Although we are not sure why the final anneal prior to the final cold rolling, and the controlled cooling of the present invention is so beneficial, we hypothesize:

that the anneal conditions the steel for cold rolling and provides an operation during which inhibitors can form; and that the slow cool to a temperature below 1700°F and/or the use of annealing temperatures in the lower part of the annealing temperature range, increase the uniformity in which the inhibitors are distributed, as essentially only ferrite phase is present in the steel at temperatures below 1700°F, contrasted to the presence of austenite and ferrite phases and different solubilities for the inhibiting elements in each phase at somewhat higher temperatures. As discussed above, the primary inhibitors are aluminum nitride, and compounds of manganese selenide and possibly manganese sulfide. No criticality is placed upon the particular annealing atmosphere. Illustrative atmosphere therefore include nitrogen; reducing gases such as hydrogen; inert gases such as argon; air; and mixtures thereof.

The following example is illustrative of several aspects of the invention.

A heat of steel was cast and processed into silicon steel having a cube-on-edge orientation. The chemistry of the heat appears hereinbelow in the Table.

TABLE

Composition (wt. %)								
C	Mn	Si	Se	S	Al	Cu	N	Fe
0.066	0.13	2.77	0.056	0.013	0.028	0.4	0.0068	Bal.

Processing for the heat involved soaking at an elevated temperature for several hours, hot rolling to a gage of approximately 93 mils, heat treating for 1 minute at 2050°F, slow cooling to 1740°F (approximately 50 seconds), air cooling to 1100°F, water quenching from 1100°F, cold rolling to a final gage of approximately 12 mils, decarburizing at a temperature of 1475°F in a mixture of wet hydrogen and nitrogen, and final texture annealing at a maximum temperature of 2150°F.

The heat was tested for permeability. A permeability of 1853 (G/O_e) at 10 oersteds was recorded.

It will be apparent to those skilled in the art that the novel principles of the invention disclosed herein in connection with specific examples thereof will suggest various other modifications and applications of the same. It is accordingly desired that in construing the breadth of the appended claims they shall not be limited to the specific examples of the invention described herein.

We claim:

1. In a process for producing electromagnetic silicon steel having a cube-on-edge orientation and a permeability of at least 1850 (G/O_e) at 10 oersteds, which process includes the steps of: preparing a melt of silicon steel; casting said steel; hot rolling said steel into a hot rolled band; subjecting said steel to at least one cold rolling; subjecting said steel to a final annealing prior to the final cold rolling; decarburizing said steel; and final texture annealing said steel; the improvement comprising the steps of carrying out said final anneal prior to the final cold rolling at a temperature of from 1400° to 2150°F for a period of from 15 seconds to 2 hours; cooling said steel from a temperature below 1700°F and above 750°F to a temperature at least as low as 500°F with a liquid quenching medium or gaseous stream and from its maximum annealing temperature to said temperature below 1700°F and above 750°F at a rate which is no faster than one wherein the steel is cooled in a static atmosphere or in a continuous processing line where there is some relative motion between the atmosphere and the steel, although the only deliberate motion is that imparted to the steel; and cold rolling the cooled steel at a reduction of at least 80%; said melt consisting essentially of, by weight, up to 0.07% carbon, from 2.60 to 4.0% silicon, from 0.03 to 0.24% manganese, at least 0.01% selenium, from 0.01 to 0.09% of material from the group consisting of sulfur and selenium, from 0.015 to 0.04% aluminum, up to 0.02% nitrogen, from 0.1 to 0.5% copper, balance iron.

2. An improvement according to claim **1**, wherein said steel is cooled from a temperature below 1600°F and above 1000°F to a temperature at least as low as 500°F with a liquid quenching medium or gaseous stream and from its maximum annealing temperature to said temperature below 1600°F and above 1000°F at a rate which is no faster than one wherein the steel is cooled in a static atmosphere or in a continuous processing line where there is some relative motion between the atmosphere and the steel, although the only deliberate motion is that imparted to the steel.

3. An improvement according to claim **1**, wherein said final anneal prior to the final cold rolling is at a temperature of from 1800° to 2125°F.

4. An improvement according to claim **3**, wherein said steel is cooled from a temperature 1600°F and above 1000°F to a temperature at least as low as 500°F with a liquid quenching medium or gaseous stream and from its maximum annealing temperature to said temperature below 1600°F and above 1000°F at a rate which is no faster than one wherein the steel is cooled in a static atmosphere or in a continuous processing line where there is some relative motion between the atmosphere and the steel, although the only deliberate motion is that imparted to the steel.

5. An improvement according to claim **1**, wherein said steel is cooled to a temperature at least as low as 500°F from a temperature below 1700°F and above 750°F with a gaseous stream.

6. An improvement according to claim **1**, wherein said steel is cooled to a temperature at least as low as 500°F from a temperature below 1700°F and above 750°F with a liquid quenching medium.

7. An improvement according to claim **1**, wherein said steel is air cooled to said temperature below 1700°F and above 750°F.

8. An improvement according to claim **3**, wherein said steel is cooled to a temperature at least as low as 500°F from a temperature below 1700°F and above 750°F with a gaseous stream.

9. An improvement according to claim **3**, wherein said steel is cooled to a temperature at least as low as 500°F from a temperature below 1700°F and above 750°F with a liquid quenching medium.

10. An improvement according to claim **3**, wherein said steel is air cooled to said temperature below 1700°F and above 750°F.

11. An improvement according to claim **1**, wherein said final anneal prior to the final cold rolling is carried out subsequent to an initial cold rolling.

12. An improvment according to claim 1, wherein said steel consists essentially of, by weight, from 0.02 to 0.07% carbon, from 2.65 to 3.25% silicon, from 0.05 to 0.20% manganese, at least 0.02% selenium, from 0.02 to 0.07% of material from the group consisting of sulfur and selenium, from 0.015 to 0.04% aluminum, from 0.0030 to 0.0090% nitrogen, from 0.1 to 0.4% copper, balance iron.

13. An improvement according to claim 1, wherein the cooled steel is cold rolled at a reduction of at least 85%.

14. An improvement according to claim 3, wherein the cooled steel is cold rolled at a reduction of at least 85%.

15. An improvement according to claim 1, wherein said final anneal prior to the final cold rolling is applied to a hot rolled band.

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