United States Patent [19]
Salsgiver et al.

[11] 3,855,019
[45] Dec. 17, 1974

[54] PROCESSING FOR HIGH PERMEABILITY SILICON STEEL COMPRISING COPPER

[75] Inventors: **James A. Salsgiver**, Sarver; **Frank A. Malagari**, Freeport, both of Pa.

[73] Assignee: **Allegheny Ludlum Industries, Inc.**, Pittsburgh, Pa.

[22] Filed: **May 7, 1973**

[21] Appl. No.: **357,973**

[52] **U.S. Cl.** .... **148/112**, 75/123 L, 148/31.55, 148/111

[51] **Int. Cl.** **H01f 1/04**

[58] **Field of Search** .... 148/112, 111, 110, 31.55, 148/113; 75/123 L

[56] **References Cited**

UNITED STATES PATENTS

2,209,686	7/1940	Crafts	148/31.55
2,867,558	1/1959	May	148/111
3,151,005	9/1964	Alworth et al.	148/111
3,159,511	12/1964	Taguchi et al.	148/113
3,287,184	11/1966	Koh	148/113
3,345,219	10/1967	Detert	148/112
3,575,739	4/1971	Fiedler	148/111
3,632,456	1/1972	Sakakura	148/111
3,671,337	6/1972	Kumai et al.	148/111
3,764,406	10/1973	Littmann	148/111
3,770,517	11/1973	Gray et al.	148/111

OTHER PUBLICATIONS

Saito, A., Effect of Minor Elements . . . in Silicon Steel, in Nippon Kinzoku, 27, (1963), pp. 191–195.

Lyman, T., Metals Handbook, ASM, Cleveland, 1948, p. 628, (TA472 A3).

Kussmann, A., et al., Gekupferter Stahl Fur . . . Transform., in Stahl und Eisen, 50, (1930), pp. 1194–1197, (T5300 S7).

Primary Examiner—Walter R. Satterfield
Attorney, Agent, or Firm—Vincent G. Gioia; Robert F. Dropkin

[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: annealing a hot rolled band of silicon steel at a temperature of from 1400° to 1700°F for a period of from 15 seconds to 2 hours; cooling the annealed hot rolled band at a rate substantially equivalent to a still air cool; and cold rolling the cooled steel at a reduction of at least 80 percent in a single cold rolling.

6 Claims, No Drawings

PROCESSING 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 percent 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 1790 to 1840 (G/O_e).

In recent years a number of patents have disclosed 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 from a processing standpoint. U.S. Pat. No. 3,287,183 which issued on Nov. 22, 1966 reveals that a steel composed of specific amounts of carbon, silicon, aluminum, sulfur and iron could be processed into a high permeability silicon steel by annealing the steel at a temperature of from 1742° to 2192°F so as to precipitate AlN, cold rolling the steel at a reduction of from 83 to 96 percent, decarburizing the steel and final texture annealing the steel. More recently, somewhat similar processing for similar alloys was disclosed in U.S. Pat. Nos. 3,632,456 and 3,636,579, which respectively issued on Jan. 4, 1972 and Jan. 25, 1972. Each of these patents refer to cooling rates following the anneal in which AlN is precipitated. U.S. Pat. No. 3,632,456 anneals a hot rolled band at a temperature of from 1382° to 2192°F depending upon its silicon content, rapidly cools the annealed band and then proceeds to subject it to at least two cold rollings. U.S. Pat. No. 3,636,579 anneals steel containing 2.5 to 4.0 percent silicon at a temperature of from 1742° to 2192°F, quenches it from said temperature to a temperature at least as low as 752°F and then cold rolls it. Another patent of interest is U.S. Pat. No. 3,159,511, which issued on Dec. 1, 1964. It discloses a process for producing silicon steel with a single cold roll, but not a process for producing silicon steels with a permeability as high as 1850 (G/O_e) at 10 oersteds. More specifically, it discloses a process wherein silicon steel is hot rolled and then directly cold rolled.

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, from steel of a particular chemistry. The method includes the steps of: annealing a hot rolled band of silicon steel at a temperature of from 1400° to 1700°F for a period of from 15 seconds to 2 hours; cooling the hot rolled band at a rate substantially equivalent to a still air cool; and cold rolling the cooled steel at a reduction of at least 80 percent in a single cold rolling. It differs from and is contradictory to the methods of heretofore referred to U.S. Pat. Nos. 3,159,511, 3,287,183, 3,632,456 and 3,636,579 in that U.S. Pat. No. 3,159,511 does not disclose a process for producing silicon steel with a permeability as high as 1850 (G/O_e) at 10 oersteds nor the step of annealing a hot rolled band at a temperature of from 1400° to 1700°F for a period of from 15 seconds to 2 hours; U.S. Pat. No. 3,287,183 discloses a minimum annealing temperature of 1742°F and not a maximum annealing temperature of 1700°F; U.S. Pat. No. 3,632,456 calls for an anneal and two cold rollings subsequent to the hot rolled band anneal, which is in fact at a temperature in excess of 1742°F for steels containing at least 2.5% Si, and does not cool the annealed hot rolled band at a rate substantially equivalent to a still air cool; and U.S. Pat. No. 3,636,579 discloses a minimum annealing temperature of 1742°F for steels containing at least 2.5% Si and a rapid cool from said annealing temperature. Moreover, the chemistry of the steel being processed in accordance with the present invention differs from that being processed in said heretofore referred to patents.

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 having, by weight, up to 0.07% carbon, from 2.60 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, and from 0.1 to 0.5% copper, casting the steel; hot rolling the steel into a hot rolled band; annealing the hot rolled hand; cold rolling the annealed hot rolled band; decarburizing the steel; and final texture annealing the steel. Also included, and significantly so, are the specific steps of: annealing the hot rolled band at a temperature of from 1400° to 1700°F for a period of from 15 seconds to 2 hours; cooling the annealed hot rolled band at a rate substantially equivalent to a still air cool; and cold rolling the cooled steel at a reduction of at least 80 percent in a single cold rolling. Preferred conditions include annealing the hot rolled band at a temperature of from 1450° to 1650°F and cold rolling at a reduction of at least 85 percent. For purposes of definition, still air cools include those wherein the steel is cooled in a static atmosphere as well as those wherein there is relative motion between the atmosphere and the steel, as in a continuous processing line, so long as there is no deliberate intention to cause the motion for cooling purposes. Moreover, for purposes of definition, all gaseous atmospheres are considered to have the same cooling effect as air. Hence, all open cools are at a rate substantially equivalent to a still air cool unless a liquid quenching medium or forced gaseous atmosphere is employed, and a forced gaseous atmosphere is one in which motion is deliberately imparted to the atmosphere for cooling purposes.

Melting, casting hot rolling, annealing, cold rolling, decarburizing and final texture annealing do not involve any novel procedures, as far as techniques are concerned, and with regard to them, the invention encompasses all applicable steelmaking procedures. Annealing of the hot rolled band at a temperature of from 1400° to 1700°F is, however, believed to be particularly beneficial in that it conditions the steel for cold rolling, provides an operation during which inhibitors can form, and most importantly, increases 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 solubilites for the inhibiting elements in each phase at somewhat higher temperature. By inhibitors, the invention primarily pertains to aluminum nitride, and manganese sulfide and/or manganese copper sulfide, which are discussed in greater detail hereinbelow. No criticality is placed upon the particular annealing atmosphere. Illustrative atmospheres therefore include nitrogen; reducing gases such as hydrogen; inert gases such as argon; air; and mixtures thereof. As to the cold rolling, it should 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, sulfur and copper. 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 sulfur are necessary as they form inhibitors which are essential to 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 sulfur to form manganese sulfide and/or manganese copper sulfide; and these compounds act so as to 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, in addition to possibly forming manganese copper sulfide, is believed to be beneficial in that it is hypothesized that copper can lower the annealing temperature, improve rollability, simplify melting and relax annealing atmospheres requirements.

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 2.60 to 3.5% silicon, a manganese equivalent of from 0.05 to 0.24% as expressed by an equivalency equation of %Mn + (0.1 to 0.25) × %Cu, from 0.01 to 0.05% sulfur, from 0.015 to 0.04% aluminum, from 0.0030 to 0.0090% nitrogen, from 0.1 to 0.3% copper, balance iron and residuals; and wherein the ratio of manganese equivalent to sulfur is in the range of from 2.0 to 4.75. The steel has its chemistry balanced so as to produce a highly beneficial structure when processed according to the present invention.

The following examples are illustrative of several aspects of the invention.

Three samples (Samples 1 – 3) of silicon steel were cast and processed into silicon steel having a cube-on-edge orientation from a heat of BOF silicon steel. The steel consisted essentially of, by weight, 0.049% carbon, 2.91% silicon, 0.094% manganese, 0.032% sulfur, 0.036% aluminum, 0.0046% nitrogen, 0.22% copper, balance iron and residuals.

Processing for the three samples involved soaking at an elevated temperature for several hours, blooming, hot rolling to a gage of approximately 92 mils, annealing at 1475°F for 1 hour in nitrogen, cooling by one of three methods, cold rolling to final gage of approximately 13 mils, decarburizing for 2 minutes at 1475°F in a mixture of nitrogen and wet hydrogen, and final annealing for 8 hours in hydrogen at a maximum temperature of 2150°F. The three cooling methods were a furnace cool, an air cool and a brine quench. Sample 1 was furnace cooled, sample 2 was air cooled and the brine quench was applied to sample 3.

Samples 1 – 3 were tested for permeability and core loss. The results of the tests appear hereinbelow in Table I.

TABLE I

Sample	Cooling	Permeability (at 10 O_e)	Core Loss (WPP at 17 KB)
1	Furnace	1651	1.27
2	Air	1860	0.785
3	Brine Quench	1902	0.708

From Table I, it is clear that samples 2 and 3 displayed very high permeabilities, that is permeabilities in excess of 1850 (G/O_e) at 10 oersteds. Sample 2 was processed in accordance with the present invention and sample 3 illustrated an embodiment of another invention claimed in copending application Ser. No. 357,974, filed concurrently herewith. Sample 2 was annealed in hydrogen for 1 hour at 1475°F, air cooled therefrom and cold rolled at a reduction of 86 percent in a single cold rolling. Similar processing was applied to samples 3 and 1 with the exception that they were respectively brine quenched and furnace cooled from their annealing temperature. Unlike samples 2 and 3, sample 1 had a relatively low permeability. It also had a core loss which was substantially higher than that for samples 2 and 3.

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 consisting essentially of, by weight, up to 0.07% carbon, from 2.60 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 and the balance iron; casting said steel; hot rolling said steel into a hot rolled band; annealing said hot rolled band; cold rolling said annealed hot rolled band; decarburizing said steel; and final texture annealing said steel; the improvement comprising the steps of annealing the hot rolled band at a temperature of from 1400° to 1700°F for a period of from 15 seconds to 2 hours; cooling said annealed hot rolled band at a rate equivalent to a steel air cool, said cool including those 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, and excluding furnace cools and those where relative motion is deliberately induced for cooling purposes and cold rolling the cooled steel at a reduction of at least 80% in a single cold rolling.

2. An improvement according to claim **1**, wherein said hot rolled band is annealed at a temperature of from 1450° to 1650°F.

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

4. An improvement according to claim **1**, wherein said steel consists essentially of, by weight, from 0.02 to 0.07% carbon, from 2.60 to 3.5% silicon, a manganese equivalent of from 0.05 to 0.24% as expressed by an equivalency equation of %Mn + (0.1 to 0.25) × %Cu, from 0.01 to 0.05% sulfur, from 0.015 to 0.04% aluminum, from 0.0030 to 0.0090% nitrogen, from 0.1 to 0.3% copper, balance iron and residuals and wherein the ratio of manganese equivalent to sulfur is in the range of from 2.0 to 4.75.

5. An improvement according to claim **4**, wherein said hot rolled band is annealed at a temperature of from 1450° to 1650°F.

6. An improvement according to claim **4**, wherein the cooled steel is cold rolled at a reduction of at least 85 percent.

* * * * *