United States Patent

[11] 3,615,919

[72] Inventors **Norman Pavlik;**
George W. Weiner, both of Pittsburgh, Pa.

[21] Appl. No. **862,524**
[22] Filed **Sept. 30, 1969**
[45] Patented **Oct. 26, 1971**

[73] Assignee **Westinghouse Electric Corporation**
Pittsburgh, Pa.

---

[54] **REACTIVE ALUMINUM NITRATE EDGE COATINGS FOR ELECTRICAL STEELS**
5 Claims, No Drawings

[52] **U.S. Cl.** **148/113,**
117/127, 148/13.1, 148/122
[51] **Int. Cl.** **H01f 1/18,**
C23d 5/00
[50] **Field of Search** 148/110,
111, 112, 113, 31.5, 31.55, 13.1, 122; 117/6, 127

[56] **References Cited**

UNITED STATES PATENTS

2,528,216	10/1950	Dunn et al.	148/113
2,828,233	3/1958	Langworthy	148/122
3,039,902	6/1962	Miller, Jr. et al.	148/113
3,132,056	5/1964	McQuade	148/31.5
3,152,930	10/1964	Foster	148/113
3,282,747	11/1966	Foster et al.	148/113
3,259,526	7/1966	Walker	148/113 X

Primary Examiner—L. Dewayne Rutledge
Assistant Examiner—G. K. White
Attorneys—F. Shapoe and R. T. Randig

ABSTRACT: A process is described for preventing the adherence of the bottom edge of adjacent convolutions of a coil of silicon steel during final box annealing to produce a cube-on-face texture. The steps include the formulation of a reactive solution of aluminum nitrate, the application of said solution to a predetermined area and drying the same prior to the application of a nonreactive insulation coating thereto.

REACTIVE ALUMINUM NITRATE EDGE COATINGS FOR ELECTRICAL STEELS

BACKGROUND OF THE INVENTION

1. Field of the Invention

The method of the present invention relates to the application of a nonreactive coating to the bottom edge of adjacent convolutions of a coil of silicon steel for the prevention of adherence of said adjacent convolutions during the final box annealing heat treatment to produce a silicon iron alloy characterized by a cube-on-face orientation.

2. Description of the Prior Art

High temperature box annealing at a temperature of about 1,200° C. is required in order to develop the texture and required magnetic properties for oriented silicon iron alloys. In the case of singly oriented silicon iron known variously as Goss texture or cube-on-edge oriented silicon iron, a coating of magnesium oxide suspended in a water slurry vehicle is usually applied to both sides of the cold rolled strip prior to winding the strip into coil configuration which coils are thereafter subjected to the high temperature box annealing. During said high temperature annealing the magnesium oxide reacts with the components of the steel forming a complex magnesium silicate glass on the steel surface. This reactive glass surface prevents the adjacent wraps of the coil from adhering to one another and yet permits the attainment of the desired crystallographic orientation within the steel, namely, that characterized as cube-on-edge texture.

On the other hand, where the desired end product is a silicon iron alloy which is characterized by a cube-on-face orientation, i.e. (100) [001]orientation, the surface coating which is applied to the adjacent convolutions of the coil must be nonreactive with the surface in order to develop the desired crystallographic orientation. In the case of the cube-on-face texture, this orientation is achieved through a surface energy phenomenon and accordingly any reaction of the surface with the coating which is applied thereto will inhibit the attainment of the desired transformation to the characterized cube-on-face texture.

In producing cube-on-face texture, it has been the practice to apply to the surface of the strip material before it is wound into coil configuration for box annealing, a coating consisting of a mixture of polyvinyl alcohol and water together with various nonreactive wetting agents. While the mixture is still wet, the strip is passed through a dust chamber usually containing high purity alumina or other nonreactive alumina which is picked up on one or both surfaces of the strip material and thereafter the solution is dried and the strip wound into coil form. In an alternative embodiment a slurry is formulated consisting of treated aluminum hydroxide powder which will form kappa alumina. A coating of the slurry of alumina, water, suspending and wetting agents is applied to both sides of the steel strip by roller coating. The coating is then dried and the strip wrapped into coil form.

It has been found, however, that even though there is a uniformity in the application of the coating, problems have been realized because of the high temperatures involved during said box annealing, said temperatures usually running of the order between about 1,100° and about 1,400° C. The coils, with coating applied to the surface thereof, are placed on edge in a furnace and one or more coils may be stacked one on top of the other. As a result of the high temperatures involved, an excessive amount of bottom edge sticking has been encountered which is believed caused by the coil wraps expanding during the annealing heat treatment together with the buckling of the coil where wide width strip is employed. Thus, the temperatures involved result in an abnormal amount of coil sagging which together with the expansion of the coils has resulted in adherence or welding with a resultant difficulty in unwrapping said coils after the transformation anneal. As a practical matter, prior to the process of the present invention it was found necessary to machine the bottom edge of each coil for a distance of a few inches in order to permit unwrapping of the coil. Since approximately 1 inch of each edge is normally slit off during the regular practice, any method which prevents said bottom edges of the adjacent convolutions of the coil from adhering to one another during final box annealing is inherently advantageous.

SUMMARY OF THE INVENTION

The process of the present invention relates to a method for preventing the bottom edges of each convolution of a coil from adhering to one another during box annealing heat treatment of the coil to produce a cube-on-face texture in a silicon iron alloy. This is accomplished by formulating a solution containing a predetermined amount of aluminum nitrate per liter of water and applying said solution to a predetermined area at the bottom edge of the strip material prior to the strip material being wound in coil form. The solution is dried at a given temperature and thereafter a nonreactive insulating coating is applied to the entire surface of the strip material prior to winding the material into coil form. The coil is thereafter placed on its edge which has been treated with aluminum nitride and given a high temperature heat treatment in order to develop the desired cube-on-face orientation within the silicon iron alloy.

An object of the present invention is to provide a method for preventing adjacent convolutions of the coil from adhering to one another during high temperature heat treatment.

A more specific object of the present invention is to provide a method for the prevention of adherence to another of adjacent convolutions of the bottom edge of a coil when the coil is set on edge and box annealed to produce a cube-on-face orientation.

Other objects of the present invention will become apparent when read in conjunction with the following description of the claims.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Since the cube-on-face grain growth arises by reason of a surface energy phenomenon occurring during a high temperature box annealing heat treatment, it becomes essential that an insulated coating be applied to the surface of the strip material, which insulating coating is nonreactive with any of the components contained within the strip material. Since, as pointed out hereinbefore, the box annealing heat treatment takes place at elevated temperatures of the order of 1,100° to 1,400° C. the coiled alloy strip material is subject to substantial thermal expansion. In addition the weight of the coil, either singly or in stack configuration, causes a certain degree of buckling during high temperature heat treatment with the result that adjacent convolutions of the coil come in contact under pressure and are subjected to edge sticking at the bottom edge of the coil when the coil is annealed in its on-edge configuration. To prevent such adherence the method of the present invention contemplates the application of a coating to the bottom edge of the strip material which coating will be reactive with the strip material and thereby prevent the adherence of adjacent convolutions of the coil from adhering to one another. For this purpose it has been found that between about 200 and about X 500 grams of aluminum nitrate dissolved in about one liter of distilled water is of a desirable concentration for such application. While variations from this range may be employed, it has been found preferred to utilize aqueous solutions having a concentration of about 200 to about 300 grams of aluminum nitrate, and excellent results have been attained using a concentration of approximately 250 grams of hydrated aluminum nitrate dissolved in 1 liter of distilled water. The solution is mixed thoroughly to dissolve the crystals. Several drops of a wetting agent may be thereafter added in order to improve the coating by the solution of the surface of the strip material.

The solution is applied to both sides of the strip edge which will form the bottom of the coil with a suitable applicator such as a felt wiper, rollers, lambs wool dauber, brush or any other convenient device which will apply enough solution to the sur-

face. It has been found that a stripe of the solution of about 1-inch wide is usually sufficient so long as the same is applied to both sides of the bottom edge of the coiled configuration. Following the application of the stripe to the iron-silicon alloy, heat is applied to dry the wet edge coating. It has been found that a temperature within the range between about 125° and about 200° C. will suffice. The aluminum nitrate solution reacts with the strip and after drying, the solution turns a characteristic brown color permitting a visual indication of coating uniformity.

Thereafter, the customary nonreactive insulating coating is applied to the entire surface of the strip or where desired, both surfaces of the strip may be coated with a nonreactive refractory material such as alumina. Good success has been achieved by first applying a water mixture containing about 4 percent by weight of polyvinyl alcohol to one or both sides of the steel strip. While the water and polyvinyl alcohol wet the surface, a dry dust consisting of finely divided alumina is applied by passing the wet strip through a cloud chamber. The polyvinyl alcohol-water-dust coating is then dried and the strip is wrapped into coil form. Alternatively, a slurry may be formulated consisting of treated aluminum hydroxide powder which forms kappa alumina. The slurry of alumina, water and suspending agents is applied to both sides of the steel sheet by roller coating to apply a thin coating of about 1 mil thickness or less. This coating is then dried and the strip is wrapped into coil form.

The coil, with the portion having the heat treated reactive coating of aluminum nitrate on the bottom edge of the coil when the coil is set on edge, is thereafter charged into a box annealing furnace and heated for the required period of time to the required temperature employing a suitable atmosphere, usually dry hydrogen, in order to develop the cube-on-face texture. During the initial period of the anneal, the polyvinyl alcohol depolymerizes and decomposes leaving substantially no carbon, whereby only alumina particles remain between the coil turns. Thereafter upon removal from the furnace, it has been found that upon removal of the insulative coating a shiny surface is present on and maintained on he steel. The application of the reactive coating to the bottom edge of the coil, has resulted in the complete freedom from adherence of the adjacent convolutions of the coil following heat treatment of the elevated temperatures.

In order to more clearly demonstrate the process of the present invention the following schedule has been employed with success:

EXAMPLE I

1. Thirty-three-inch wide cold rolled strip was employed, said strip being of a finish gauge thickness of about 0.012 inch. The cold rolled strip was degreased using tricholorethylene and dried.

2. A solution of a concentration of 250 grams of $A1(NO_3)_3 \cdot 9H_20$ per liter of water was mixed and agitated until all of the crystals were dissolved. A 1 inch wide stripe of less than a 1 mil thickness of this solution was applied to both sides of the bottom of the strip only.

3. The edge coated strip was dried at 150° C. strip temperature producing a brown layer where the aluminum nitride solution was applied and the strip was thereafter wrapped into coil form.

4. After cooling to room temperature, a coating of water containing 4 percent by weight of polyvinyl alcohol was applied to one side of the steel sheet. While the coating of the polyvinyl alcohol-water mixture was wet, a dry dust consisting of alumina of a fineness of less than 300 mesh was applied by passing the wet strip through a cloud chamber. The polyvinyl alcohol-water coating was then dried by heating to 150° C. and the strip wrapped into the coil.

5. Coil was set on edge with the aluminum nitrate layer at the bottom and box annealed at 1,200° C. for 12 hours.

6. Following cooling to room temperature the coil was unwound, scrubbed, and edge trimmed. No difficulty was encountered in unwinding the coil following the box annealing.

EXAMPLE II

Step 4 of example I is substituted for by applying to the sheet steel of a slurry of five parts of 300 mesh alumina in 100 parts of 4 percent polyvinyl alcohol-water suspension, with 1/10 of 1 percent of a wetting agent.

It has been found that the edge coating of aluminum nitrate reacts with the sheet surface at the box annealing temperature and forms a surface which prevents the coil edge form sticking despite the expansion, buckling and weight of the coil.

Since the aluminum nitrate coating does not contaminate or inhibit transformation to the cube-on-face texture except possibly at the area of application, great success has been achieved with attaining the maximum transformation to cube-on-face texture. Since approximately 1 inch of the edge of the strip to which the reactive aluminum nitrate coating was applied is normally slit in regular practice, no additional waste has been encountered, yet is has been possible to unwind the coils without an expensive machining operation to remove the adhering adjacent convolutions of the coil.

While it has been disclosed hereinbefore that the stripe is applied to both sides of the surface of the coil of the bottom edge of the coil only, only one side of the strip need be coated for preventing the edge adherence. In addition, where the coils may be stacked on top of one another it may be desirable to coat both the bottom and the top edge of the strip with the aluminum nitrate solution. In either event such alternatives are contemplated within the scope of the present invention.

We claim:

1. In a method of producing coils of iron-silicon alloy for producing a cube-on-face texture and in which a strip of the alloy is coated with a nonreactive refractory sheet separator, and the coated strip is wound into coil form, placed on edge and annealed at a temperature of about 1,100° to 1,400° C., the improvement comprising applying a solution containing between about 200 and about 500 grams of aluminum nitrate per liter of water, over a predetermined edge portion area to at least one side of the strip and drying said strip before the application of the refractory sheet separator whereby the bottom edges of each convolution of the coil are prevented from adhering to one another during said annealing.

2. The method of claim 1 in which said solution is applied in a stripe extending about 1 inch wide from the bottom edge of the coil.

3. The method of claim 1 in which the solution applied to the predetermined area is dried at a temperature within the range between about 125° and about 200° C.

4. The method of claim 1 in which the solution is prepared to have an aluminum nitrate concentration within the range between about 200 grams and about 300 grams per liter of water.

5. In the method of preventing the bottom edges of the convolutions of a coil of an iron-silicon alloy from adhering to one another after annealing at a temperature of about 1,200° C. for producing a cube-on-face texture and in which at least one side of each convolution of the coil is treated with a mixture of polyvinyl alcohol, water and alumina for preventing adhesion of the respective convolutions during annealing, the improvement comprising, making a solution containing between about 200 grams and about 300 grams of aluminum nitrate per liter of water, applying a stripe extending about 1 inch from the bottom edge of the coil to each side of each convolution of said bottom edge and drying said solution at a temperature within the range between about 125° and about 200° C. before applying said water-alcohol alumina mixture thereto.

* * * * *