

1

2,875,113

METHOD OF DECARBURIZING SILICON STEEL IN A WET INERT GAS ATMOSPHERE

Elmore J. Fitz, Lanesboro, Mass., assignor to General Electric Company, a corporation of New York

No Drawing. Application November 15, 1957
Serial No. 696,616

7 Claims. (Cl. 148—113)

The present invention relates to magnetic silicon steel for electrical uses, such as in transformers, motors and other electromagnetic apparatus, and more particularly to an improved process of producing decarburized silicon steel.

The silicon steel material to which the present invention relates normally contains about 3% silicon, but the silicon content may vary between 1% and 4%.

In addition, the carbon content of silicon steel ingots used as starting material for making electrical steel of the type herein concerned is generally about 0.05% or less, with values of 0.020 to 0.030% carbon being typical. It is generally accepted that the lower the carbon content of processed silicon steel, the better are its ultimate magnetic properties. Even the above relatively small percentages of carbon will cause the watt loss of the final magnetic strip to be undesirably high if it is not further reduced in the course of processing the metal. The maximum amount of carbon tolerable from the standpoint of low watt losses is now considered to be about 0.006%. Recent work has indicated that if increasing core loss in transformers, for example, with the passage of time is to be completely avoided, the carbon content of the core steel must be held to 0.002% or less.

The conventional method of processing silicon steel material of the above type is to subject it to a series of rolling and heat treating (annealing) steps for reducing the material to strip form of desired gauge while removing undesired impurities and internal strains therefrom. In an intermediate heating step in such process, the rolled material is usually subjected to a heat treatment of about 950° C. for softening the strip to facilitate further rolling and to relieve the strains therein. Normally, after the desired gauge has been obtained, the strip is subjected to a decarburizing treatment to reduce the carbon content to a minimum. This treatment, in the past, has been carried out in decarburizing atmospheres at a temperature of about 800° C., which was considered by the prior art to afford the most effective removal of the carbon in the shortest possible time. Temperatures higher than that range and particularly above 900° C. were found, using prior processes, to result either in less carbon removal or require longer heating periods, or both. As a consequence, the decarburizing treatment has conventionally been carried out in a stage separate from the intermediate strain-relief anneal in which temperatures well above 900° C. are most effective.

It is an object of the invention to provide an improved process of producing decarburized silicon steel which is simpler, shorter and produces more effectively decarburized silicon steel than prior known methods.

It is another object of the invention to provide silicon steel material having substantially reduced carbon content as compared to that made by conventional processes.

It is a further object of the invention to provide a rapid process of decarburizing silicon steel at temperatures above 900° C., and a process of treating steel strip

2

wherein the decarburizing step may be carried out together with the strain-relief heat treatment normally used in processing silicon steel.

In accordance with the invention, it has been found that when the silicon steel strip is thoroughly pre-cleaned, as hereinafter described, and thereafter subjected to a heat treatment in the presence of a wet pure inert gas, such as nitrogen, and wherein the temperature is in the range of 875° C. to about 1025° C., the carbon content of the final silicon steel material is reduced to a level not heretofore attained by the usual mill processes, and such reduction is effected in an extremely short decarburizing process.

Various types of reducing and oxidizing atmospheres have been used in the prior art in carrying out decarburizing processes, but the previously used gases have not been fully satisfactory in that in certain cases the carbon level reached was not sufficiently low, the time required to effect adequate decarburization was too long, the results obtained were non-uniform, or for other reasons.

In certain of the atmospheres previously used, oxygen or hydrogen was incorporated, but it has been discovered in connection with the present invention that the presence of either oxygen or hydrogen in the decarburizing atmosphere actually serves to hinder effective removal of carbon from the steel to very low levels, contrary to the teachings of the prior art. In other cases, gases such as carbon dioxide have been incorporated in decarburizing atmospheres, but such carbon-bearing gases have been also found to prevent the attainment of extremely low levels of carbon content, presumably due to an equilibrium established between the carbon-bearing gas and the steel which prevented further removal of the carbon once equilibrium had been reached.

It appears that the decarburizing action produced in accordance with the invention is effected chiefly by the moisture component of the present gas mixture and that the remaining gas component or components serve merely as diluents for the water vapor and should be inert to the metal and its constituents.

Accordingly, it is of particular significance in the invention that reactive gases such as oxygen and hydrogen, and carbon-bearing gases such as carbon dioxide, are entirely avoided, and to this end a pure inert diluent gas such as nitrogen is used as a vehicle for the decarburizing water vapor. While nitrogen is a particularly suitable inert gas, especially from an economic standpoint, other inert types of gases such as argon, helium, krypton, neon and the like, or mixtures thereof, may alternatively be used where appropriate.

The proportion of water vapor used with the nitrogen or equivalent gas is not critical in the present invention, it having been found that substantially the same results in terms of carbon removal are obtained whether the atmosphere is saturated with moisture or contains only a relatively small proportion thereof. For example, successful decarburization has been accomplished with dew points as low as -40° F. However, it is preferred to operate with dew points above 35° F., since the process is then less sensitive to slight variations in surface preparation. Raising the dew point above 35° F. does not improve the efficiency of the process but does offer the practical advantage of making it possible to add the desired water vapor to the pure inert gas by passing the latter through a water bath at room temperature, e. g., about 70-80° F.

It is of importance to ensure that the steel surface is thoroughly cleaned prior to the decarburizing treatment, especially for the purpose of removing any scale which may result from a prior hot-rolling stage. For this purpose, the steel may be shot-blasted, pickled in

an acid solution, e. g., hydrochloric and hydrofluoric acid mixture, and then washed in water. The cleaning action should be sufficiently thorough to provide a clean, bright surface on the steel. It is also desirable to remove any grease on the surface, and this may be accomplished, for example, by passing the material through a conventional vapor degreaser prior to its entrance into the decarburizing chamber.

Decarburization as carried out by the present process is particularly effective in the temperature range of 875° to 1025° C., which is in contrast with prior art teaching that best decarburization is achieved at a temperature of less than 875° C. Since the optimum temperature range for the stress-relief anneal is about 925° to 955° C., the present process is eminently suited for combination with the stress-relief anneal treatment in processing the silicon steel. The time required for maximum removal of carbon using the present process is of the order of 10 minutes or less.

A series of tests was conducted to compare the effects of various known decarburizing furnace atmospheres with that of the present invention. In these tests, groups of ten samples each of identical silicon steel strip 14 mils thick were all initially subjected to a cleaning step which involved pickling in an acid solution, degreasing and washing, after which each sample was heat treated for 10 minutes at 925° C. Each group of ten samples was treated in a different one of four types of atmospheres consisting of the following: (a) line nitrogen, containing 0.2–0.3% oxygen; (b) pure nitrogen (line nitrogen with oxygen removed); (c) air; (d) substantially pure hydrogen. Water vapor was incorporated in each of the atmospheres, the dew point being 78–80° F.

In addition, a further group of ten samples of identical material, not pre-cleaned as above described, was also heat treated at the same temperature in a pure wet nitrogen atmosphere of the above-mentioned moisture content.

Analysis of carbon content of these samples after the decarburizing treatment showed the following average results for the samples treated in each type of atmosphere:

Atmosphere:	Carbon content, percent
Line nitrogen-----	.002
Air-----	.003
Line hydrogen-----	.014
Pure nitrogen-----	.001

The additional samples which had not been pre-cleaned and which had been subjected to a pure wet nitrogen atmosphere showed a carbon content of 0.003%.

Carbon contents below .002% are difficult to determine accurately by the analytical techniques presently used, and there is good reason to believe that the true value for those samples subjected to the pure wet nitrogen atmosphere of the present invention is substantially below the .001% value shown. Of the ten samples tested in this group, nine showed a carbon content of .001% and one showed .002%. The results obtained with the samples pre-cleaned and decarburized in pure wet nitrogen in accordance with the invention thus showed marked improvement over the decarburizing processes using other types of atmospheres, and the reduction thereby afforded in terms of reduced magnetic aging losses of the silicon steel makes the present process of considerable practical value. This is of particular significance in view of experimental evidence which shows that the rate of magnetic aging increases substantially above .002% carbon content.

The results of the above tests also show that the scale left on samples subjected to the decarburization process hinders removal of the carbon to an appreciable

extent, and that the removal of this scale and other impurities by the pre-cleaning procedure described is essential to consistently attain maximum removal of carbon from the steel.

In a typical process which may be used in practicing the invention, a silicon steel ingot containing about 3% silicon and .02% carbon is initially hot rolled to about .085 inch and after removal of the scale and other impurities by a pre-cleaning step, it is cold rolled to about .028 inch. Then the steel strip is subjected to a combined strain-relief anneal and decarburizing treatment in a pure wet nitrogen atmosphere at a temperature of about 925 to 955° C. for about four minutes. Thereafter, the decarburized strip is cold rolled to a gauge of .012–.014 inch. The material thus produced is ready for further refining and grain growth processes well known in the art.

In the above process, the pre-cleaning step may be carried out immediately after the hot rolling stage as described, or it may be deferred until just prior to the combined strain-relief and decarburizing treatment.

By virtue of the invention silicon steel strip may be made with virtually the minimum detectable carbon content, and it may be effectively produced by the use of a decarburizing heat treatment having a relatively wide range of temperature as compared to the temperatures previously considered optimum in the prior art for reducing carbon to minimum levels. Of particular advantage is the provision by the invention of the combination of decarburizing and annealing steps using elevated temperatures of well above the 900° C. level which is optimum for annealing purposes, thus enabling the elimination of the usual final decarburizing step, and in addition providing for carbon contents lower than obtained by any of the conventional decarburizing treatments in equivalent periods of time.

While the present invention has been described with reference to particular embodiments thereof, it will be understood that numerous modifications may be made by those skilled in the art without actually departing from the scope of the invention. Therefore, the appended claims are intended to cover all such equivalent variations as come within the true spirit and scope of the invention.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. The method of treating silicon steel which comprises pre-cleaning the silicon steel, and heating the silicon steel at a temperature of 875–1025° C. in an atmosphere of pure wet inert gas.

2. The method of treating silicon steel which comprises pre-cleaning the silicon steel to provide a clean, bright surface thereon, and heating the silicon steel at a temperature of 875–1025° C. in an atmosphere of pure inert gas containing water vapor.

3. The method of treating silicon steel which comprises pre-cleaning the silicon steel to provide a clean, bright surface thereon, and heating the silicon steel at a temperature of 925° C.–955° C. in an atmosphere of pure nitrogen containing water vapor sufficient to provide a dew point of at least about –40° F.

4. The method of producing decarburized silicon steel which comprises hot rolling silicon steel material to an intermediate gauge, cleaning the hot rolled silicon steel material to provide a clean, bright surface thereon, cold rolling the thus treated silicon steel material to a further reduced gauge, subjecting the silicon steel material to a temperature of 925° C.–955° C. in an atmosphere of pure inert gas containing water vapor to provide a combined decarburization and stress-relief anneal, and cold rolling the thus treated material to final gauge.

5. In the process of producing magnetic silicon steel, the steps of pre-cleaning the silicon steel to provide a clean, bright surface thereon, and thereafter subjecting the cleaned silicon steel to a combined strain-relief and

5

decarburizing treatment which comprises heating the silicon steel at a temperature of 925° C.-955° C. in an atmosphere of pure inert gas containing water vapor sufficient to provide a dew point of at least about -40° F.

6. Electrical silicon steel as made by the method defined in claim 1.

7. Electrical silicon steel as made by the method defined in claim 4.

6**References Cited in the file of this patent****UNITED STATES PATENTS**

1,358,810	Yensen	Nov. 16, 1920
2,287,467	Carpenter et al.	June 23, 1942
2,534,141	Morrill et al.	Dec. 12, 1950

OTHER REFERENCES

Yensen: Transactions, American Institute of Electrical Engineers, vol. 43, 1924, page 166.