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PRODUCTION OF SILICON STEEL SHEET STOCK HAVING THE PROPERTY OF HIGH SURFACE RESISTIVITY

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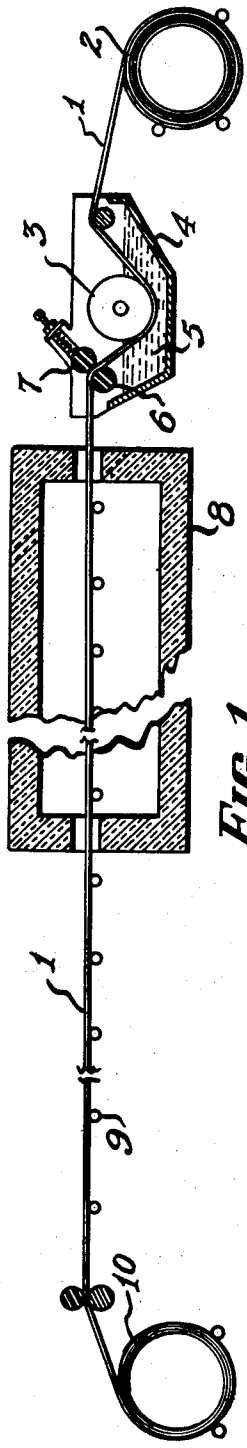


FIG. 1.

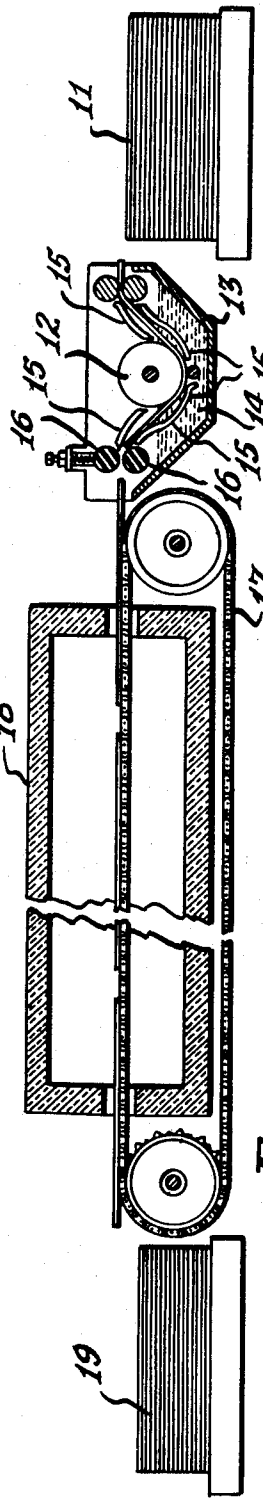


FIG. 2.

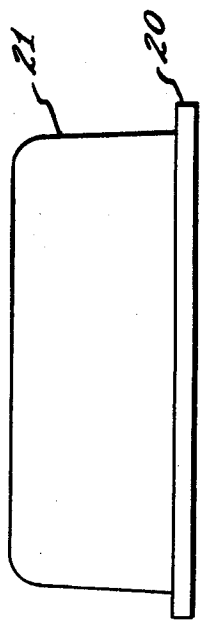


FIG. 3.

INVENTOR.
CARL E. GIFFORD.
BY Allen & Allen
ATTORNEYS.

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PRODUCTION OF SILICON STEEL SHEET STOCK HAVING THE PROPERTY OF HIGH SURFACE RESISTIVITY

Carl E. Gifford, Zanesville, Ohio, assignor to Armco Steel Corporation, a corporation of Ohio

Application October 3, 1945, Serial No. 620,055

18 Claims. (Cl. 148—6.15)

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This is a continuation-in-part of my copending application of the same title, Serial No. 400,094, filed June 27, 1941, now abandoned.

The primary object of my invention is the production of silicon steel sheet stock (by which I mean silicon steel of sheet gauge, either in the form of coils of strip of indefinite length or in the form of individual sheets), which stock is characterized by high surface resistivity, and the production from such stock of laminated cores having a high interlamination resistivity as well as an excellent space factor.

It is another object of my invention to attain these results by a process and treatment which is simple, requiring little additional labor, equipment and materials, and which is comparatively inexpensive in cost.

The need for high interlamination resistivity in magnetic cores such as are used in power transformers has long been recognized, to minimize the losses due to eddy currents. It has been the practice in the art to coat transformer laminations with insulative substances. Both organic and inorganic insulative coatings have been proposed; but the application of such coatings is a matter of inconvenience and expense. Reliance upon the insulative value of an imposed substance, for example, silicate of soda or a core plate enamel, leads to the use of relatively thick coatings to be certain of the minimum required resistance. The nature of imposed coatings also is frequently such as to prevent or interfere with after treatments for the silicon steel, such as heat treatments and the like. Difficulty has been experienced with some coatings because they tend to be attacked by the oil in the transformers.

It is an object of my invention to provide an insulative surfacing which does not have any of these defects.

It is an object of my invention to provide an insulative coating which is capable of commercial use not only for the highest grades of power transformers but for other electric apparatus as well.

It is an object of my invention to provide an insulative surfacing which has a number of incidental advantages, as will be set forth hereinafter, besides providing a high interlamination resistivity and a high space factor.

It is an object of my invention to provide an insulative surfacing for silicon steel which can be controlled as to its various characteristics to meet the needs of various types of service for electrical steels, all as will hereinafter be set forth.

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These and other objects of my invention which will be set forth below or will be apparent to one skilled in the art upon reading these specifications, I accomplish by those procedures and in those treated articles of which I shall now describe exemplary embodiments.

While my treated sheet stock has a very high and uniform surface resistivity, and while my treatment of the sheet stock does not diminish the space factor thereof in any significant degree, it may be understood that by the term "high resistivity" herein I mean an interlamination resistivity in the assembled core of at least 1 ohm per square centimeter per lamination; and by "good space factor" I mean a space factor of at least 90%, or preferably 95% or better, both the resistivity and the space factor being determined when the laminated core is subjected to a pressure of 50 lbs. per square inch.

Reference is made to the accompanying drawings, in which:

Figure 1 is a diagrammatic elevational view of an apparatus for treating strip.

Figure 2 is a diagrammatic elevational view of an apparatus for treating sheets.

Figure 3 is a diagrammatic representation of an annealing base and cover.

I have found that by chemical and thermal treatments, involving the formation of a resistive coating, presumably by the interaction of constituents of the base metal and a chemical which may be thinly and evenly applied from a solution, I am enabled to produce a surfacing on my sheet stock which not only is of high and dependable resistivity, but also is exceedingly thin and exceedingly uniform. These results cannot be accomplished, so far as I am aware, in any treatment for insulation where an insulative substance is imposed as such upon the surfaces of the sheet stock.

Modes of forming upon the surfaces of metal various coatings which are believed to be or comprise phosphates have been current in the art of producing iron and mild steel sheets and articles, and in the art of producing galvanized or other metal coated sheets and articles. As applied to the former, the result is a passivating of the surface, rendering it less susceptible to rust and corrosion. As applied to the latter, the result is a fitting of the surface for the immediate reception of paints, enamels and the like.

These treatments have generally comprised holding the articles in a solution or electrolyte for a sufficient length of time to permit the desired extent of chemical attack. The solution

or electrolyte was usually a dilute solution of phosphoric acid or metal phosphates or both, frequently with control agents and accelerators such as nitrates, metallic salts other than phosphates, and the like. In some instances, more concentrated solutions have been made into pastes with neutral filler materials, coated upon the surfaces of the metallic articles, allowed to dry (during which time chemical action occurred), and then cleaned from the surfaces.

It has hitherto been suggested that silicon steel laminations be treated in phosphating solutions like those referred to above, but with the object of producing upon the silicon steel laminations coatings of high resistivity. The results, however, have been disappointing. As applied to silicon steel, the phosphating solutions have not built up coatings having the required characteristics; and in particular, it has not been possible to secure resistivities, as measured by the standard tests, as great as one ohm per square centimeter per lamination. In addition, these former procedures have a number of serious disavantages. The material must be left in contact with the solution for a sufficient length of time to permit the desired chemical attack, and it must then be rinsed or otherwise cleaned. This makes for a time-consuming operation and involves the use of relatively elaborate and costly equipment for the steps mentioned. Yet again, in respects other than a lack of resistivity, the coatings have been unsatisfactory.

I have, however, discovered ways in which thoroughly satisfactory resistive coatings, completely responsive to the objects of this invention, may be formed upon the surfaces of silicon steel or other ferro-magnetic materials by simple and rapid procedures and with a minimum of equipment.

Briefly, in the practice of my invention, I subject silicon steel sheet or strip stock to a brief passage through a solution of phosphoric acid, followed by a calendering or wiping action to make sure that the solution is distributed upon the surfaces in a uniform manner. Without any necessary intermediate drying, and in actual practice without any drying at all, the sheet or strip stock is then subjected to a controlled heat treatment as hereinafter more fully explained. With a satisfactory distribution of chemical upon the surfaces of the sheet or strip, the qualities of the final coating are determined by the nature of the heat treatment and can be widely varied, all as hereinafter taught.

The solution which I prefer to use is a solution of phosphoric acid. It is preferably used as a concentrated solution; but the degree of concentration may be dependent, within practical limits, upon the thickness of the film of solution left upon the metal surfaces. Thus the same quantity of residual phosphoric acid may be derived from a thicker film of less concentrated solution or from a thinner film of more concentrated solution. In general, I employ solutions ranging from about 7.25% of free phosphoric acid based on the weight of the solution to those containing 50% by weight of the free acid, preferring ordinarily those containing about 35% of free acid.

It is essential that the film of solution be gauged or calendered upon the surface of the stock, and while there are various ways of doing this, I prefer to pass the stock through rubber covered rolls which are adjustable as to spacing. Here again it will be understood that a continuous material, such as stock rolled and maintained as a strip, or strip stock formed by welding sheets together

end to end, can be more accurately and carefully calendered than individual sheets. Individual sheets are thus likely to have a somewhat less uniform and generally thicker residual coating of solution, and this may be compensated in part at least by varying the strength of the solution.

The solution-gauging rolls need not be made of rubber, since other materials will also be effective. Under some conditions more uniform and controlled results will be obtained if the rolls are lightly grooved.

The manner of initial application of the solution is not a limitation. The surfaces of the sheet stock (which will be in a cleaned condition, i. e. free of oils, greases and scale) may be sprayed or dipped or swabbed with the solution. I have found it more convenient merely to pass the stock through a narrow bath of the solution by causing it to be submerged beneath a roll turning in such a bath. Upon emerging from the bath the sheet or strip stock is passed immediately through solution-gauging rolls and thence into a furnace.

Referring to Figure 1 which is diagrammatic in character, I have shown a strip 1 of silicon steel being withdrawn from a coil 2 in a standard decoiler, and passing beneath roll 3, turning in a pan 4 of phosphoric acid solution 5. Rubber wiping rolls are indicated at 6 and 7, the strip 1 passing between them and thence into a gas fired furnace 8 beyond which it may be cooled as at 9 and recoiled as at 10.

A similar apparatus may be employed for sheets as diagrammatically indicated in Figure 2. The sheets are taken individually by the operators from a stack 11 and are passed beneath a roll 12 turning in a tank 13 of phosphoric acid solution 14. Guides 15 assist in determining the path of movement of the sheets and in delivering them to solution-gauging rolls 16. A conveyor 17 immediately carries them through a short furnace 18 at the far end of which they are stacked as at 19.

For certain purposes, the recoiled strip or the stacked sheets produced as set forth above may subsequently be annealed, if desired, in a controlled atmosphere, Figure 3 being diagrammatically illustrative of an annealing base 20 and an annealing cover 21 in which a controlled atmosphere may be maintained.

As indicated, I prefer to use a simple solution of phosphoric acid. Advantageous results can sometimes be obtained by adding magnesium oxide to the phosphoric acid in quantities to be wholly dissolved by the acid. Other materials, such as those frequently used for phosphate-coating iron or mild steel, may be added to the phosphoric acid, including accelerators, nitrates, and the like; but these are unnecessary in my practice. Metal phosphates may be employed to some extent in lieu of phosphoric acid. I have not, however, found such additions or substitutions advantageous, and in many instances they are detrimental. My best results have been obtained with phosphoric acid alone or with phosphoric acid solutions in which magnesia has been dissolved, and, by way of example, it is my practice to replace any phosphoric acid solution which through a prolonged period of use has become contaminated with iron.

It will be understood, however, that the passage of the strip or sheets through the bath of phosphoric acid solution is rapid, that the solution is continuously being carried from the baths upon the surfaces of the sheets and strip, and that therefore the baths have to be renewed from

time to time with fresh solution to compensate for this carry-out. The passage of the sheets and strip through the bath is so rapid in my operation that, contrary to usual practices in the phosphate-coating of iron and steel, I do not believe any substantial degree of chemical attack occurs in the bath. Hence it is not a feature of my operation to leave the sheet or strip stock in contact with a bath of electrolyte until chemical action has occurred. On the contrary, a continuous furnace is employed and the stock is passed immediately into the furnace without drying. Under the influence of the furnace heat, drying occurs very rapidly, as will hereinafter be more fully discussed, and I believe that the principal chemical reactions occur in the furnace.

The quantity of solution left upon the surfaces of the silicon steel in the coating step and hence the weight of the final formed coating may be varied through a rather wide range, keeping in mind that if the coatings are too thick they may adversely affect space factor, while it is possible to make coatings so thin as to be deficient in resistance. Solely as a guide to the skilled worker, and without limitative intent, I may say that stock produced commercially by me shows, upon chemical analysis, an average of .006 ounce of phosphorus per square foot of coated surface.

An entirely suitable solution can be made, for example, by diluting commercial concentrated phosphoric acid (containing around 75% phosphoric acid by weight) with an equal volume of water. Using such a solution to attain the coating just mentioned, it will be evident that about 2.59 pounds or .265 gallon of the solution will be required to treat each 1000 square feet of silicon steel surface. If, by way of example, a silicon steel .003 inch in thickness is being coated, 86.5 pounds of the solution or 8.83 gallons will be required to coat one ton of the steel to produce a coating containing .006 ounce of phosphorus per square foot of coated surface.

I have found that when a film of solution is imposed upon silicon steel as taught above, a heat treatment becomes necessary to produce satisfactory results. Without a heat treatment, the coating is quite likely to assume and retain a mucilaginous or tacky condition because phosphoric acid is hygroscopic. No coating is formed having sufficient strength or cohesiveness, and none having sufficient resistivity.

I have discovered that various effects occur upon the application of heat at different temperatures, and also that under certain circumstances the nature of the atmosphere during the heat treatment affects the results obtained. I have found that the results are classifiable into three groups of conditions, obtainable respectively in the temperature range of substantially 400° F. to 700° F., in the temperature range of substantially 700° to 1000° F., and at temperatures above 1000° F., up to and including annealing temperatures for developing magnetic properties.

The varying results obtainable have different utilities; and some consideration of the requirements of different forms of service is helpful at this point.

Where the strip or sheet stock is to be formed by punching into laminations for transformer cores or the parts for rotating machinery, the matter of die life is important. The stock should not contain or be covered with any highly abrasive material which would be productive of excessive wear on the dies, i. e., the stock should have a good die life. This is generally true of

silicon steel used in the form of separate laminations, excepting where the laminations are very large and are sheared out instead of being punched. Any powdery substance which might build up on the dies must be avoided. A powdery material which is so loose in character as to be productive of a cloud of dust around the punch press is undesirable. On the other hand, the sticking together of punched laminations during re-annealing is not generally a problem, since the pressure on the laminations during re-annealing is seldom sufficiently high to cause trouble, even when high annealing temperatures are used.

With the production of modern types of highly directional silicon steel having very great permeabilities in the rolling direction, the making of wound transformer cores has become important. Die life is of minor consequence in the production of stock for such cores because the material is slit, not punched. The dusting problem remains, not only because dust in the atmosphere is highly undesirable, but also because a coating of loose, chalky and friable character on the stock is likely to interfere with plastic bonding in the production of wound cores where a bonding step is practiced. A coating which in itself will fuse and stick during re-annealing is undesirable because it may give rise to a condition interfering with or preventing the proper impregnation of the wound core with the plastic bonding agent.

I modify my heat treatments in accordance with these considerations as follows:

A heat treatment of the filmed stock at temperatures of substantially 400° to 700° F. is productive of a coating which is satisfactory from the standpoint of resistivity but has a characteristic which may be termed "chalky." This characteristic may vary in intensity; but the coating, while of fair durability, is of such character that a chalky appearance may readily be observed upon passing the thumb nail over it or upon using some other scraping instrument under gentle pressure. The dusty substance is likely to collect upon punching dies and cause trouble, as well as to contaminate the atmosphere of the factory or work room. The nature of the coating is such that it will fuse under high temperatures and hence may stick in wound cores.

The dust difficulty may be eliminated with materials of this character by scrubbing the sheet or strip stock. This removes the friable or chalky portion of the coating while leaving a more desirable portion which still has sufficient interlamination resistivity for the purpose set forth. I have found that sheets treated at temperatures higher than about 700° F. tend to lose their flatness, but that temperatures between substantially 400° and 700° F. are not objectionable in this respect. Hence, in spite of the fact that scrubbing as an additional operation somewhat increases the cost, my recommended procedure where sheets must be treated individually includes heating the sheets within the said range of substantially 400° to 700° F. followed, if required, by scrubbing.

It is possible to minimize the dusting difficulty by employing thin initial films of solution, and hence to avoid scrubbing; but it is very difficult to calender the thinner films on sheets. If the sheets are to be joined by welding to make strip stock or if cold rolled strip stock is being manufactured, I prefer operating in the second temperature range where the passage of strip stock

through a furnace under slight tension at higher temperatures prevents loss of flatness.

When a strip stock bearing a film of solution formed as indicated is subjected to temperatures lying substantially between 700° and 1000° F. another type of result is obtained. The coating becomes very durable, tightly adherent and cohesive throughout, and entirely loses the chalky or dusty character mentioned above. Abrasion of the coating no longer produces the powdery appearance; and the coating assumes a darker and more uniform gray color. I believe that the coating fuses under these temperatures to a very much more homogeneous condition. This is suggested by the appearance of the coating. Also, whereas in heat treating the stock at temperatures between substantially 400° and 700° F. attention must be given to the rate of heating to prevent the formation of a bubbly or frothy appearance in the coating, the rate of heating becomes very much less important when the temperature is carried into the second range which I have mentioned. While it is still advisable to avoid so rapid a rate of heating as to produce an exaggerated frothiness in the coating or to blow it from the surfaces of the strip stock, sporadic frothiness or bubbling of the coating disappears as the temperature rises, again indicating fusion.

A coating formed within the second temperature range gives good die life, very high resistivity and great durability. The stock is thus excellent for punching. The coating is, however, still fusible at temperatures ordinarily employed for strain annealing. This may be found objectionable for certain types of wound core structures to be impregnated with a bonding agent after annealing. In the case of transformer laminations or stamped parts for electrical machinery, the sticking tendency is of no consequence for reasons already given.

Excellent results are obtained in this temperature range by heating the strip stock to a temperature around 800° F.

When the temperature is carried above 1000° F. yet another phenomenon occurs. The coating changes in some fashion so as not to be refusible at temperatures suitable for strain annealing. Where the heating occurs in an oxidizing atmosphere, the coating takes on a dark reddish color believed by me to be due to the presence in it of oxides of iron. These oxides, however, do not impair the resistivity of the coating, which remains very high; but they do adversely affect die life. This is a distinct disadvantage for materials from which laminae or parts are to be punched; but for wound cores it is not a disadvantage because the material for such cores is slit or sheared. Heating stock coated as described to temperatures above 1000° F. will produce materials excellent for wound core uses.

If it be desired to gain the aforesaid condition of infusibility in the coating without the production of oxides, this may be accomplished by heat treating the stock in a nonoxidizing or reducing atmosphere. A continuous furnace, equipped with gas seals at its entrance and exit ends and also equipped with a protective cooling hood in which the metal is maintained in the same or equivalent atmosphere until it has cooled below the oxidation temperature, may be kept filled with a neutral or reducing atmosphere, and the result obtained in this way.

When heating magnetic stock to high temperatures, care must be taken to avoid an impairment of its magnetic qualities. With certain kinds of stock, a rapid heating to a temperature, say, of 1250° F., followed by a rapid cooling, may be detrimental to magnetic qualities. But the practice of my invention does not preclude the use of a subsequent box anneal for the development of ultimate magnetic qualities. My materials should be given a preliminary heat treatment when they are later to be box annealed, and when the nature of the box anneal is such that sticking of stacked sheets or wound coils is expected and is to be avoided, then the preliminary heat treatment will preferably carry the stock to a temperature of 1000° F. or higher. This may be done in a non-oxidizing atmosphere as set forth above. My coatings, formed as taught and heat treated at 1000° F. or higher (whether or not an oxidizing atmosphere is used), provide efficient annealing separators for high temperature annealing.

I do not wish to be bound by theory as to the precise chemical nature of my coating. Presumably, the phosphoric acid reacts chemically with the iron of the base metal, for the most part, in the furnace. Apparently, however, there is a distinct chemical difference between a coating formed by low temperature drying on the surface of the stock and a coating which has been heated at least to a temperature of around 400° F. Whether there is a chemical or only a physical difference between a coating which has been heat treated at from substantially 400° to 700° F. and one which has been heat treated at substantially 700° to 1000° F. is not known to me. The loss of the property of fusibility at strain annealing temperatures above 1000° F. would seem to indicate a chemical difference between such coatings and those heated to lower temperatures.

In the preferred practice of my invention I employ short continuous furnaces, maintaining them at the desired temperatures, and send my sheet or strip materials through these furnaces at such speeds as will permit them to come up to furnace temperature.

I have indicated that the surfaces of the stock should be clean prior to my treatment. I know of no other requirement. My coating procedure operates equally well on mechanically cleaned surfaces and on surfaces chemically cleaned as by pickling. My procedures are applicable to silicon steels, by way of example, which have been previously annealed in dry nitrogen (see Davidson and Mahlie Patent No. 2,229,642) and to silicon steels which have previously been decarburized in wet hydrogen (see Carpenter and Jackson Patent No. 2,287,467), each of which treatments may result in a certain amount of silica on or near the surfaces of the stock. My treatment may even be applied to silicon steel bearing a glassy coating produced as taught in the patent to Carpenter, Bell and Heck No. 2,385,332, issued September 25, 1945. Heat treatments at the lower two temperature ranges are not recommended when a glassy coating is already present; but at temperatures above 1000° F., an excellent coating is formed. Where the atmosphere is oxidizing, a slight red color may appear; but the presence of the silica glass largely prevents oxidation. In a neutral or reducing atmosphere, no oxidation occurs. While the glassy coating referred to is ordinarily satisfactory for interlamination resistivity, my process applied to such materials will correct defective stock, or may be employed to produce extraordinarily high resistivities where required, as in transformers having high voltages per turn.

While my treatments have been described in connection with silicon steel, they are applicable to other magnetic materials including pure iron, nickel iron alloys with or without silicon, and the like. While I have described my procedures as applied to sheet or strip stock, it will be apparent that they may be applied to other forms of silicon steel, e. g. laminations.

A pickling step will remove the insulative coatings formed by my process; but should it be found necessary for any reason to pickle a batch of the coated stock, the operations herein taught may thereafter be repeated.

My treated stock is especially valuable in that its surface resistivity persists in spite of handling, stamping, shearing, winding into wound cores, and annealing, as set forth above.

Silicon steel stock in sheet or strip form or in the form of laminations or cores, after having been given my treatment, is much less susceptible to rusting. This is of particular value where such materials are to be stored for long periods of time or transported over great distances or under unfavorable circumstances. Also, materials treated in accordance with my teachings have a high utility for heat-resistant uses and will exhibit a much longer life than ordinary materials in such uses.

The resistive surfaces formed by my treatments do not deteriorate in use; they are not attacked by transformer oils and the like; and though exceedingly thin, have a very high surface resistance.

Modifications may be made in my invention without departing from the spirit thereof. Having thus described my invention in certain exemplary embodiments, what I claim as new and desire to secure by Letters Patent is:

1. A process of treating ferrous magnetic sheet gauge stock to give it a high surface resistivity which comprises treating the clean surfaces of the ferrous stock with a water solution of phosphoric acid containing substantially between 7% and 50% by weight of the acid, metering the quantity of residual solution remaining on the surfaces of the stock to a thin, non-flowing film of substantially uniform thickness, and subjecting the stock to an open annealing heat treatment in an atmosphere non-reducing to phosphates at a temperature of from substantially 400° F. to and including annealing temperatures above 1000° F. for developing magnetic properties, to the extent of causing the phosphoric acid to react with the iron of said ferrous magnetic sheet stock and form a resident insulative phosphate coating thereon having a resistance of at least 1 ohm per square centimeter at a pressure of 50 pounds per square inch and a space factor of at least substantially 90%.

2. The process of claim 1 in which the solution is applied by passing the stock briefly through a bath of said solution, and is metered by passing the stock through solution-gauging rolls.

3. The process of claim 1 in which the solution is applied by passing the stock briefly through a bath of said solution, and is metered by passing the stock through solution-gauging rolls, and then directly, without drying, into a furnace wherein the stock is heated as aforesaid.

4. A process of treating ferrous magnetic sheets to produce high surface resistivity thereon, which comprises passing said sheets with clean surfaces through a water solution of phosphoric acid containing substantially between 7% and 50% by weight of the acid, metering the film of solution remaining on the surfaces of the sheets by passing them through solution-gauging rolls, and then passing the sheets without drying into an open annealing furnace with an oxidizing atmosphere wherein the said sheets are heated to temperatures substantially between 400° and 700° F.

5. A process of treating ferrous magnetic sheets to produce high surface resistivity thereon, which comprises passing said sheets with clean surfaces through a water solution of phosphoric acid containing substantially between 7% and 50% by weight of the acid, metering the film of solution remaining on the surfaces of the sheets by passing them through solution-gauging rolls, then passing the sheets without drying into an open annealing furnace with an oxidizing atmosphere wherein the said sheets are heated to temperatures substantially between 400° and 700° F., and thereafter scrubbing the surfaces of the said sheets.

6. A process of treating ferrous magnetic sheet-gauge strip stock to give it a high surface resistivity, which comprises continuously and rapidly passing the strip stock through a bath of a water solution of phosphoric acid containing substantially between 7% and 50% by weight of the acid, thence to solution-gauging rolls to meter residual solution on the surfaces of the strip stock to a uniform film, and thence without drying through an open annealing furnace with an atmosphere non-reducing to phosphates and heating the said strip stock rapidly to a temperature substantially between 700° and 1000° F. whereby to form upon the surfaces of the strip stock a non-chalky, highly resistive coating of great durability.

7. The process of claim 6 wherein an oxidizing atmosphere is maintained in the said furnace.

8. A process of treating ferrous magnetic sheet-gauge strip stock to give it a high surface resistivity, which comprises passing the stock briefly through a bath of a water solution of phosphoric acid containing substantially between 7% and 50% by weight of the acid, metering the residual film left upon the surfaces of the said strip stock by passing it between solution-gauging rolls, thence carrying the stock without drying into and rapidly through a continuous furnace and heating it rapidly in an atmosphere non-reducing to phosphate to a temperature between about 1000° F. and a higher temperature up to and including annealing temperatures for developing magnetic properties, whereby to form upon the surfaces of said strip stock a resistive phosphate coating, non-fusible at temperatures for reannealing.

9. The process claimed in claim 8 in which the atmosphere of the said furnace is an oxidizing atmosphere, the resultant coating having a reddish hue.

10. The process claimed in claim 1 in which the temperature to which the stock is heated is a temperature suitable for annealing to develop magnetic characteristics therein and is a temperature in excess of substantially 1000° F.

11. The process claimed in claim 1 in which the temperature to which the stock is heated is a temperature suitable for annealing to develop magnetic characteristics therein and is a temperature in excess of substantially 1000° F., and in which the heat treatment is carried on in an oxidizing atmosphere.

12. The process claimed in claim 1 in which

the temperature to which the stock is heated is a temperature suitable for annealing to develop magnetic characteristics therein and is a temperature in excess of substantially 1000° F., and in which the heating is carried on in a non-oxidizing atmosphere.

13. Silicon steel sheet-gauge stock having on its surface a thin, uniform, tightly adherent coating consisting essentially of the reaction products of iron and a material chosen from a class consisting of phosphoric acid and phosphoric acid in which magnesia has been dissolved, said reaction products produced at a temperature not less than 400° F., the iron in said reaction products being derived substantially entirely from the iron in said sheet-gauge stock, said coating having a high resistivity of at least one ohm per square centimeter per lamination at a pressure of 50 pounds per square inch, said coating having a powder-free surface of a non-hygroscopic character, said coating being of a thickness to contain about .006 ounce of phosphorus per square foot of coated surface, and being heat-fusible.

14. Silicon steel sheet-gauge stock having on its surface a thin, uniform, tightly adherent coating consisting essentially of the reaction products of iron and a material chosen from a class consisting of phosphoric acid and phosphoric acid in which magnesia has been dissolved, said reaction products produced at a temperature not less than about 700° F., the iron in said reaction products being derived substantially entirely from the iron in said sheet-gauge stock, said coating having a high resistivity of at least one ohm per square centimeter per lamination at a pressure of 50 pounds per square inch, said coating being in a fused condition, and non-hygroscopic, and being of a thickness to contain about .006 ounce of phosphorus per square foot of coated surface.

15. Silicon steel sheet-gauge stock having on its surface a thin, uniform, tightly adherent coating consisting essentially of the reaction products of iron and a material chosen from a class consisting of phosphoric acid and phosphoric acid in which magnesia has been dissolved, said reaction products produced at a temperature of at least 1000° F. in an oxidizing atmosphere, the iron in said phosphate being derived principally from the iron in said sheet-gauge stock, said coating having a high resistivity of at least one ohm per square centimeter per lamination at a pressure of 50 pounds per square inch, said coating having the characteristic of infusibility at strain annealing temperatures, having a reddish hue, and having a thickness to contain about .006 ounce of phosphorus per square foot of coated surface.

16. Silicon steel sheet-gauge stock having on its surface a thin, uniform, tightly adherent coating consisting essentially of the reaction products of iron and a material chosen from a class consisting of phosphoric acid and phosphoric acid in which magnesia has been dissolved, said reaction products produced at a temperature of at least 1000° F., the iron in said phosphate being derived principally from the iron in said sheet-gauge stock, said coating having a high resistivity of at least one ohm per square centimeter per lamination at a pressure of 50 pounds per square inch, said coating having the characteristics of infusibility under strain annealing temperatures and having a thickness to contain about .006 ounce of phosphorus per square foot of coated surface.

17. Silicon steel sheet-gauge stock having on its surface a thin, uniform, tightly adherent coating consisting essentially of the reaction products of iron and phosphoric acid in which magnesia has been dissolved, said reaction products produced at a temperature not less than 400° F., the iron in said reaction products being derived substantially entirely from the iron in said sheet-gauge stock, said coating having a high resistivity of at least 1 ohm per square centimeter per lamination at a pressure of 50 pounds per square inch, said coating having a powder-free surface of a non-hygroscopic character, said coating being of a thickness to contain about .006 ounce of phosphorus per square foot of coated surface, and being heat fusible.

18. Silicon steel sheet-gauge stock having on its surface a thin, uniform, tightly adherent coating consisting essentially of the reaction products of iron and phosphoric acid in which magnesia has been dissolved, said reaction products produced at a temperature not less than about 700° F., the iron in said reaction products being derived substantially entirely from the iron in said sheet-gauge stock, said coating having a high resistivity of at least 1 ohm per square centimeter per lamination at a pressure of 50 pounds per square inch, said coating being in a fused condition, and non-hygroscopic and being of a thickness to contain about .006 ounce of phosphorus per square foot of coated surface.

CARL E. GIFFORD.

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