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COMPOSITION AND METHOD FOR INCREASING SURFACE RESISTIVITY OF SILICON STEEL

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This invention relates to the production of silicon steel sheet stock used in the manufacture of electrical equipment and more particularly to production of such stock possessing high surface resistivity.

For efficient electrical performance, the individual laminations of silicon steel sheet forming the cores of power transformers and the like must be insulated one from the other. In many instances the necessary insulation can be provided by treating the surfaces of the stock prior to fabrication with a suspension of an inert material such as diatomaceous earth or the like dispersed in a water solution of a binding agent, sodium silicate being commonly used for the latter purpose. This simple method, termed "inorganic core plating," however cannot be used on stock intended for the manufacture of oil-cooled cores, e. g., the cores of large transformers. It has been found in such applications that the sodium silicate film breaks down by reacting with organic acids formed in the oil by oxidation thereof during service. The breakdown not only destroys the insulating character of the film but produces a heavy sludge which ultimately clogs the oil circulating system causing overheating and damage to the transformer. The substitution of phosphoric acid for sodium silicate in the core plating mixture has been suggested as a possible solution to these difficulties, however this has proven unsuccessful in commercial practice since the resulting core plate tends to be deliquescent, thus deteriorates rapidly and makes handling and storage of treated stock extremely difficult. This situation has forced resort to siccative insulating varnishes. While these provide a much greater degree of insulation, e. g., surface resistivity in excess of 100,000 ohms/cm.²/strip at 1,000 p. s. i. is commonly attained with varnish whereas resistivities of several thousand ohms are unusual in inorganic core plating operations, they are considerably more expensive both as to initial cost and cost of application. Moreover, such varnishes can only be applied after all thermal treatment of the stock is complete, thus adding further to the cost of manufacture.

It is an object of the present invention to overcome the aforementioned deficiencies of inorganic core plating and to provide an improved inorganic core plate which is not only non-deliquescent and free from sludge-forming tendencies but one which will provide a silicon steel stock having a surface resistivity of the order of 100,000 ohms/cm.²/strip at pressures of 1,000 p. s. i. and above with a lamination factor not more than one percent lower than before the coating is applied.

The foregoing and other objects, which will be apparent as the description proceeds, are achieved through the use of a core plate slurry comprising a substantially neutral aqueous solution of mono- and di-ammonium phosphates, and silica preferably diatomaceous earth, ground to a particle size not greater than 5 microns. The present invention contemplates applying a thin film of this slurry to the surfaces of silicon steel stock, then heating the coated stock to dry and cure the slurry coating in situ.

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The slurry may be applied in any of several ways; roller coating is particularly advantageous since this facilitates regulation of the amount of slurry applied, particularly when handling silicon steel stock in strip form.

While the exact manner of application is relatively unimportant, the composition and character of the slurry is critical. We have found that solutions of ammonium phosphate salts having a pH of 7 are most desirable; such can readily be prepared using a mixture of 9:1 parts by weight of mono-ammonium phosphate to di-ammonium phosphate respectively. Some variation of course is permissible, however ammonium phosphate salt mixtures which produce a pH less than 5 or more than 9 must be avoided. Slurries more acidic than pH 5 produce deliquescent coatings; while those more basic than pH 9 do not bind well and troubles with sludging are not avoided. The concentration of the ammonium phosphate salts must also be controlled. The salts must comprise not less than 5% of the total weight of the slurry, otherwise the coatings are not properly bonded. On the other hand, more than 30% of the salts result in blistering and flaking of the coating during the curing operation. In addition, we have found that the slurry must contain at least 5% by weight of diatomaceous earth. While up to 40% of this agent may be used, the optimum amount appears to be about 35%. However, this varies somewhat with the method of application, the quantity of ammonium phosphate and pH of the solution. While other forms of silica, e. g., silica flour, may be used in compounding the slurry of the present invention, we prefer diatomaceous earth since this material is relatively pure silica in a readily comminutable form. As previously mentioned, it is essential that the particle size of the silica be reduced to 5 microns or less. The importance of particle size in achieving high surface resistivities cannot be overemphasized. Slurries subjected to ball milling to reduce the particle size of the silica to below the above specified maximum will consistently produce material possessing a surface resistivity in excess of 100,000 ohms/cm.²/strip at 1,000 p. s. i. after the final anneal and with a satisfactory lamination factor, i. e., a lamination factor not more than one percent lower than for uncoated laminations; whereas the maximum surface resistivity coupled with a satisfactory lamination factor which can be attained with slurries not treated to reduce the particle size thereof is of the order of 15,000 ohms/cm.²/strip at this pressure.

While any slurry composition falling within the above defined ranges will produce our results, to illustrate operations of our invention we prefer a mixture prepared from:

Parts by weight	
Mono-ammonium phosphate.....	10
Di-ammonium phosphate.....	90
Diatomaceous earth.....	600
Water	1000

These ingredients are mixed and the mixture ball-milled for 4-5 hours, which procedure reduces the particle size of the diatomaceous earth to between ½ and 2½ microns and produces a slurry, having the consistency of a heavy cream, which is readily applicable by conventional roller coating apparatus. A uniform film of this slurry is applied to both sides of the silicon steel stock and the coated stock is then heated to about 420° F. for 2-4 minutes to dry and partially cure or react the phosphates of the slurry with the iron of the surfaces of the stock. The temperature of the stock is then raised to about 1400° F. for 2 minutes to complete the curing and develop maximum resistivity. The foregoing is preferred practice. Any temperature between 400° F. and about 1600° F. can be used provided sufficient time is allowed for curing.

Cured coatings are stable even at high temperatures provided the heating is conducted in an oxidizing atmosphere. Drying and curing may be combined in a single heating step, however the coatings of the present invention are non-deliquestent even after low temperature drying and thus may be stored and handled without damage prior to the curing operation. This permits certain operational advantages to be realized, for example, the stock can be coated and dried in strip form, the required laminations sheared and punched therefrom, and the curing accomplished in the subsequent strain-relief annealing operation. When practiced in this manner the present invention completely eliminates the cost of the intermediate high temperature curing step required heretofore. In either mode of practice however, the present method consistently produces ferrous magnetic stock having surface resistivity in excess of 100,000 ohms/cm.²/strip at pressures of 1,000 p. s. i. or more. Further, the stock is completely inert to transformer oil and thus avoids the sludging troubles encountered heretofore with inorganic core plates.

While we have shown and described certain specific embodiments of our invention, we do not wish to be limited exactly thereto, but only by the scope of the following claims.

We claim:

1. The method of treating silicon steel stock and the like to produce upon the surfaces thereof a tightly adherent coating characterized by being non-hygroscopic and inert to transformer oil and having a surface resistivity in excess of 100,000 ohms/cm.²/strip at pressures of 1000 p. s. i. which comprises applying to the surfaces of said stock a thin uniform film of a slurry containing 5 to 40% by weight of silica having a maximum particle size of 5 microns dispersed in an aqueous solution of ammonium phosphates having a pH of between 5 and 9 and present in amounts sufficient to constitute 5 to 30% by weight of the slurry, and then heating the coated stock to a temperature of 400 to 1600° F. to dry and cure the coating to the surfaces of said stock.

2. The method of treating silicon steel stock and the like to produce upon the surfaces thereof a tightly adherent coating characterized by being non-hygroscopic and inert to transformer oil and having a surface resistivity in excess of 100,000 ohms/cm.²/strip at pressures of 1000 p. s. i. which comprises applying to the surfaces of said stock a thin uniform film of a slurry containing 5 to 40% by weight of diatomaceous earth having a maximum particle size of 5 microns dispersed in an aqueous solution of 5 to 30% by weight of a mixture of 9 parts by weight of mono-ammonium phosphate and 1 part by weight of di-ammonium phosphate, and then heating the

coated stock to a temperature of 400 to 1600° F. to dry and cure the coating to the surfaces of said stock.

3. The method of treating silicon steel stock and the like to produce upon the surfaces thereof a tightly adherent coating characterized by being non-hygroscopic and inert to transformer oil and having a surface resistivity in excess of 100,000 ohms/cm.²/strip at pressures of 1000 p. s. i. which comprises applying to the surfaces of said stock a thin uniform film of a slurry containing 5 to 40% by weight of diatomaceous earth having a maximum particle size of 5 microns dispersed in an aqueous solution of 5 to 30% by weight of a mixture of 9 parts by weight of mono-ammonium phosphate and 1 part by weight of di-ammonium phosphate, and then heating the coated stock to a temperature of about 400° F. for sufficient time to dry and partly cure the coating and then subsequently heating the stock to a temperature of about 1400° F. to complete the curing of the coating to the surfaces of said stock.

4. A composition for the inorganic core plating of silicon steel stock and the like comprising an aqueous slurry having a pH between 5 and 9 consisting of 5 to 40% by weight of silica having a maximum particle size of 5 microns, 5 to 30% by weight of ammonium phosphate and the balance water.

5. A composition for the inorganic core plating of silicon steel stock and the like comprising an aqueous slurry consisting of 5 to 40% by weight of diatomaceous earth having a maximum particle size of 5 microns, 5 to 30% by weight of the mixture consisting of 9 parts by weight mono-ammonium phosphate and 1 part by weight di-ammonium phosphate, and the balance water.

6. A composition for the inorganic core plating of silicon steel stock and the like comprising an aqueous slurry consisting of

	Parts by weight
Mono-ammonium phosphate-----	10
Di-ammonium phosphate-----	90
Diatomaceous earth-----	600
Water -----	1000

said diatomaceous earth having a maximum particle size of 5 microns.

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