United States Patent Office

3,297,493

Patented Jan. 10, 1967

3,297,493

PROCESS FOR PROVIDING AN IMPROVED PHOSPHATE INSULATING COATING ON FERROUS MAGNETIC MATERIAL

Heinz Blum and Hans-Joachim Edler, Frankfurt am Main, Germany, assignors to Hooker Chemical Corporation, Niagara Falls, N.Y., a corporation of New York

No Drawing. Filed Mar. 1, 1963, Ser. No. 262,222
Claims priority, application Germany, Mar. 20, 1962, M 52,192
1 Claim. (Cl. 148—6.15)

The present invention broadly relates to the formation of electrically nonconductive coatings on ferrous substrates and more particularly is directed to a process for forming an improved electrically insulating phosphate coating of high resistivity on magnetic sheet material which is suitable for use in the manufacture of laminated cores of the type employed in transformers, electric motors, electric generators, and the like.

A variety of coating compositions and processes are presently in use for forming insulating coatings on the surfaces of ferrous magnetic sheet materials facilitating their use in the manufacture of laminated cores employed in various electrical components. Phosphate-type coatings have been found particularly suitable for this purpose due to their excellent insulating characteristics and high resistivity. However, phosphate coatings of the types heretofore known are usually characterized as being relatively fragile resulting in the formation of dust during the handling of the coated sheet as well as during the forming, stamping, blanking and cutting operations employed for the manufacture of the laminated cores. The dusting characteristics of phosphate coatings contribute not only to undesirable working conditions but also cause an accumulation of dust on the tools and machinery frequently resulting in a reduction of their useful operating life.

It is accordingly, a principal object of the present invention to provide an improved phosphate coating and method of making the same which overcomes the dusting problem associated with phosphate coatings of similar type heretofore known.

Another object of the present invention is to provide a process for forming an improved phosphate coating which not only substantially eliminates the dusting problem but further increases the electrical insulating characteristics and heat conductivity characteristics of the coating providing for improved laminated cores made therefrom.

Still another object of the present invention is to provide an improved process for forming inorganic nonmetallic phosphate insulating coatings which is simple and economical to control and operate and which provides a coating which substantially enhances the processing versatility and use in which the coated ferrous magnetic sheet material can be employed.

The foregoing and other objects and advantages of the present invention are achieved by applying a layer-type phosphate coating on the surface of a ferrous sheet material and thereafter impregnating the pores of the resultant porous phosphate coating with a controlled quantity of an oily substance such that the resultant oil-impregnated coating has a substantially dry surface.

While the present invention is applicable to phosphate coatings applied to any one of a variety of metal substrates, it is particularly applicable for forming improved insulating coatings on ferrous magnetic sheet material of the type employed in electrical apparatus such as the cores of transformers, motors, generators, etc. Ferrous magnetic materials which possess the requisite magnetizable qualities for making laminated cores include silicon steel,

common iron, nickel-iron alloys, and the like. The laminations of which the cores are comprised are conventionally stamped, blanked, punched, or otherwise cut from large sheets, strips, or coils of strip material of indefinite lengths which, prior to such cutting operations have been provided with a phosphate coating in accordance with the practice of the present invention. It is also contemplated within the scope of the present invention that the phosphate coating can be applied directly to the surfaces of the individual laminar pieces after they have been cut from the ferrous magnetic sheet or strip material.

Inorganic nonmetallic phosphate coatings which possess satisfactory electrical insulating characteristics and are of the requisite porosity for absorbing oil in the pores thereof in a manner and quantity so as to achieve the benefits of the present invention, include phosphate coatings of the reactive or so-called "coating-type" including zinc phosphates, manganese phosphates, ferrous phosphates, in addition to pseudo-coating phosphates such as calcium phosphates and magnesium phosphates, as well as mixtures thereof. The foregoing metallic phosphates can be employed singly or in combination to provide the requisite adherent coating of the desired thickness on the surface of the metal strip. Of the foregoing coating phosphates, solutions of zinc phosphates or solutions comprising predominantly zinc phosphates modified by smaller proportions of the other coating phosphates such as iron or calcium phosphates, for example, constitute the preferred coating compositions.

Reactive or coating type phosphates of the aforementioned type are conventionally applied in the form of an aqueous solution containing a phosphate ion concentration calculated as PO_4 usually ranging from about 0.3% up to about 3% by weight. In addition to the metallic phosphate salts which may comprise metallic salts of, for example, dihydrogen phosphates, pyro-phosphates, metaphosphates, and the like, the solution can also contain suitable oxidizing or accelerating agents such as nitrates, chlorates, nitrites, sulphites, peroxides, including organic oxides such as nitro organic compounds such as, for example, nitrobenzine sulphonate, and the like as well as mixtures thereof. The oxidizing or accelerator agent is usually employed in concentrations ranging from a few hundreds of a percent such as about 0.05% to amounts up to about 3% by weight of the solution. The particular amount of accelerator agent employed depends on the type and concentration of the metal phosphate salts used as well as the oxidizing characteristics of the accelerator agent in addition to the temperature and time conditions under which the solution is applied to the metal substrate to achieve the requisite coating thickness.

The coating type phosphate solutions are controlled within an acidity range usually ranging from a pH of about 1.8 up to a pH of about 3.0. The specific acidity of the solution is established by the composition and concentration of the phosphates and accelerator agents employed and may be further adjusted by including small proportions of free phosphoric acid so as to provide the requisite acidity to achieve optimum coating results.

The phosphate solution can be applied to the surface of the metallic sheet or strip in any manner well known in the art such as, for example, by immersion, dipping, flooding, spraying, etc., for a period of time sufficient to deposit a coating of the requisite thickness. The solutions are conventionally applied at temperatures from about 120° F. up to about 190° F. requiring immersion times of from about 2 to 10 minutes and spray times usually ranging from about 30 seconds up to about 2 minutes.

It has been found that to provide the requisite electrical insulating characteristics and resistivity as required between laminations of a laminated core, the coatings should have a thickness of at least about 2 microns and preferably a thickness ranging from about 6 microns up to about 15 microns. Accordingly, the phosphate solution and conditions of applying the phosphate solutions are controlled so as to deposit a phosphate layer having a thickness within the aforesaid range.

After the magnetic sheet material has been coated with a phosphate coating and thereafter dried, the resulting dry phosphate coating is of a dull gray appearance and of a substantially porous nature in which it is susceptible to excessive powdering during subsequent handling and as a result of mutual abrasion between adjacent sheets as well as during the stamping, cutting, and forming operations. In accordance with the practice of the present invention, the porous phosphate coating is impregnated with an oily substance which is absorbed within the pores thereof eliminating the dusting tendency of the coating during handling of the coated sheet as well as increasing its electrical insulating and heat conductivity characteristics. The impregnation of the pores of the phosphate coating cannot be achieved by conventional oiling practices as heretofore employed to increase the coating's corrosion protection since it is an important feature of this invention that the oil be applied in a manner so that no excess thereof is present on the exterior surface of the coating. Accordingly, the application of oil is controlled in a manner as hereinafter set forth so that the resultant oil-impregnated coating has a "substantially dry" surface.

A substantially dry surface as herein set forth and as employed in the subjoined claims as defined as one that is sufficiently oil free so that when conventional blotting paper is pressed against the impregnated coating for a period of several seconds, no visible absorption of oil by the blotting paper takes place. At the same time, however, the surface of the phosphate coating is not completely free of oil to the extent that a water wetting test shows a surface that is free of grease. The presence of an excess quantity of oil on the surface of a coating as opposed to a dry surface condition as hereinabove defined as undesirable and unsuitable for the purposes of the present invention since such excess oil causes the formation of gaseous products which produce a porosity when molten aluminum is cast around the assembled laminated cores producing an unacceptable assembly. On the other hand, the oil which is absorbed within the pores of the coating does not contribute to the formation of such gaseous products and no imperfection in the aluminum casting is obtained.

In order to achieve good penetration and absorption of the oily substance within the pores of the phosphate coating, an oil of a relatively low viscosity is preferably employed such as a viscosity below about 10° (Engler degrees) measured at 20° C. Conventional petroleum oils such as for example, spindle oils, are particularly suitable for impregnating the pores of the coating. Oils of vegetable origin and synthetic oils such as silicone oils can also be satisfactorily employed but conventional spindle oils are preferred. When metals such as aluminum for example, are cast in a molten condition around the assembled laminated core, the heating of the core to an elevated temperature necessitates that oils of relatively high boiling point be employed. Conventionally, oils having a boiling point above about 300° C. are preferred and have been found satisfactory for withstanding the heat effects during such a casing operation. Lower boiling point oils under such conditions are undesirable since their increased volatlity results in the formation of gaseous products during the casting operation which causes porosity in the surrounding cast metal imparting a corresponding reduction in its conductivity and the further possibility of short circuiting a rotor employing the laminated core or reducing the tightness of laminated core within the surrounding casting of a stator assembly.

The oil cannot be satisfactorily applied to the coating by simple immersion due to the uncontrolled surface quantities or residual oil deposited on the surfaces of the coating which does not provide the requisite dry surface conditions as hereinabove described. In order to achieve substantially complete penetration and absorption of the oily substance within the pores and to simultaneously render the resultant impregnated phosphate surface substantially dry, the oil can be applied to the phosphate coating in the form of an extremely fine sized spray and preferably by employing an aqueous emulsion of oil in which the coated strip or sheet is immersed. The application of the oil employing an aqueous emulsion is controlled such that the residual emulsion film on the phosphate coating contains a quantity of oil corresponding to that which can be satisfactorily absorbed by the pores of the phosphate coating. The use of an aqueous oil emulsion provides the advantage in that when the phosphate coating is applied by immersion or spraying of the phosphate solution on the surfaces of the metal strip, the resultant wet metal strip can be directly immersed in the oil water emulsion followed by a drying step during which the water is removed and the oil in the residuary emulsion film remains and penetrates into the pores of the coating.

In order to further illustrate the method comprising the present invention, the following example is provided. It will be understood, however, that the example is presented only for illustrative purposes and is not intended to be limiting of the scope of the present invention as set forth in the subjoined claims.

Example I

A cold rolled silicon steel grip containing 0.05% carbon, 0.05% silicon and the balance iron having a thickness of 0.5 millimeter (0.0197 inch) was coated in a phosphate solution having the following composition:

Ingredient:	Grams per liter
P_2O_5	6.9
NO_3	13.5
Zn	6.7
Fe	2.4
Water	Balance

The steel strip was immersed in the phosphate solution maintained at a temperature of about 55° C. having a composition as set forth in the foregoing table for a period of 10 minutes. At the expiration of the treatment time, the coated sheet was rinsed with cold water and was thereafter immersed in an aqueous oil-water emulsion containing 40 grams per liter of a petroleum spindle oil and 4 grams per liter of a suitable emulsifying agent of any of the types well known in the art and the balance water which was held at a temperature of 80° C. The coated strip was continuously drawn through the heated emulsion at a speed-time relationship so that the strip became heated to the temperature of the bath at about 80° C. and was thereafter drawn out carrying on the surfaces thereof a residuary emulsion film in an amount of approximately 40 grams/square meter (3.7 grams per square foot). The residuary emulsion film was thereafter dried by infra-red radiation, for example, whereby the residuary water in the pores and on the surface of the coating derived from the phosphating treatment and the water rinsing, as well as the water in the emulsion was evaporated leaving the residuary oil which penetrated into the pores of the coating. At the completion of the oil coating treatment, the dry oil impregnated coated sheet material had an appearance of a slightly darker color of dried phosphate coatings which are produced in the same manner but which were not impregnated with the oily substance.

While it will be apparent that the preferred embodiments herein illustrated are well calculated to fulfill the objects above stated, it will be appreciated that the invention is susceptible to modification, variation, and change without departing from the proper scope or fair meaning of the subjoined claims.

What is claimed is:

A process for forming an improved electrically insulating phosphate coating on ferrous magnetic sheet material which comprises the steps of immersing said sheet material in a phosphating solution containing a phosphate salt selected from the group consisting of zinc phosphates, manganese phosphates, iron phosphates, calcium phosphates, magnesium phosphates, and mixtures thereof for a period of time sufficient to deposit a porous adherent phosphate layer thereon, rinsing said phosphate coating with water, and thereafter immersing the rinsed and coated said sheet material in an aqueous emulsion containing an oil selected from the group consisting of petroleum oils, vegetable oils, synthetic oils, and mixtures thereof having a viscosity less than about 10 Engler degrees and a boiling point greater than about 300° C., removing said sheet material from said emulsion leaving a residuary emulsion film thereon containing oil in an amount up to that which can be absorbed within the pores of said coating, and thereafter drying said sheet material to evaporate substantially all of the residuary water therefrom and effecting penetration of said oil and impregnation of said pores by said oil, leaving a coating on the surface which is substantially dry and oil free.

References Cited by the Examiner

UNITED STATES PATENTS

1,247,668	11/1917	Gooding	148—6.15 X
1,850,726	3/1932	Pfalzgraff.	
1,997,550	4/1935	O'Leary	148—6.15 X
2,191,435	2/1940	Ballard et al.	148—6.15
3,114,661	12/1963	Palm	148—6.15 X

ALFRED L. LEAVITT, *Primary Examiner.*

RALPH S. KENDALL, *Examiner.*