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PRODUCTION OF MAGNETIC MATERIAL

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5 Claims. (Cl. 117—100)

This application is a division of United States patent application Serial No. 139,000, filed April 26, 1937, now Patent No. 2,232,352, entitled "Production of magnetic material and of magnetic cores therefrom."

It is common to produce magnetic cores for intermediate and high frequency purposes from finely powdered magnetic material such as iron or ferromagnetic alloys, by compressing the particles by means of a binding agent to form a solid material. For the purpose of avoiding conductive relative contact between the said particles, various methods have been proposed according to which these particles are provided, prior to squeezing, with an insulating film. Thus, for example, it has been described to superficially attack the particles chemically by treating iron particles with a phosphoric acid solution or a solution of a phosphoric acid compound, a ferric phosphate film being formed on the said particles due to the reaction with the iron.

According to the invention, in contradistinction thereto, a film of a phosphoric acid metal compound is mounted on the magnetic particles without these particles assisting genetically in the film formation, which can be ensured by precipitation of the phosphoric acid metal compound from a dispersion which practically does not attack the magnetic particles.

The invention offers the advantage that shape and dimensions of the magnetic particles remain unaltered since for the formation of the insulating film no material of the magnetic particles is chemically transformed. This is particularly conductive to uniform manufacture since the quantity of insulating material per particle of the magnetic material can be controlled without any difficulty to a sufficiently accurate extent, which is substantially more difficult with chemical attack of the surface since the rate and the uniformity of the chemical attack per particle can only be controlled with difficulty. The fact that in accordance with the invention no gas development as a result of chemical reaction arises is also conductive to uniformity.

The invention is consequently particularly adapted for use with magnetic particles the dimensions of which are in the order of magnitude of a few μ, the thickness of the insulating films to be obtained not exceeding 20 mμ.

Since in accordance with the invention the magnetic particles have been provided with a thin insulating film, the magnetic powder thus obtained can be given any shape by compression. In order to increase the mechanical strength of the magnet core a binding agent may be used with advantage in a manner known per se after the insulating film is mounted. For this purpose, use may be made, for example of a synthetic resin which may be capable of being hardened. In the use of a resin capable of being hardened, even distribution of the magnetic particles in the final product may be assisted by grinding the material after mixture with the resin, by next giving it at ordinary temperatures the desired shape and by then heating it to cause it to harden.

Example I

1 kg. of magnetic powder is introduced into 1 litre of a 2 normal solution of aqua-ammonia containing 60 milliequivalents of zinc ion and 60 milliequivalents of phosphate ion. The excess of aqua-ammonia is then removed by boiling while shaking continuously so that all the zinc phosphate present in the solution is precipitated on the surface of the particles. After being filtered and dried, the powder is squeezed into the desired shape at a pressure of about 5000 kg./cm.^{2}.

Example II

1 kg. of magnetic powder is intimately mixed with about 100 cm.^{3} of a 2 normal solution of aqua-ammonia containing 30 milliequivalents of zinc ion and 45 milliequivalents of phosphate ion per 100 cm.^{3}. The paste thus obtained is kneaded while evaporating the excess of aqua-ammonia and water, which may be effected while heating to a low extent or in vacuo at ordinary temperatures. The material is then completely dried and ground to form a powder which is mixed with an alcohol solution containing 60 grams of a hardening phenolformaldehyde condensation product. After volatilization of the solvent the powdering operation is renewed and the powder is then squeezed at 150° C. into the desired shape, it being possible to apply a squeezing pressure of 1500 kg./cm.^{2}.

In order to avoid the presence of other electrolytes which would affect the properties of the core, the ammoniacal zinc phosphate solution may be obtained by newly precipitated zinc hydroxide being dissolved in a concentrated solution of aqua-ammonia, by a dilute phosphoric acid solution being next added and by the solution being then given the desired concentration by dilution.

Example III

1 kg. of magnetic powder is intimately mixed with about 100 cm.^{3} of a weakly-acid ferric phos-

phate solution containing 30 milliequivalents of ferri-ion and 30 milliequivalents of phosphate ion per 100 cm.^{3}. The paste is evaporated and dried, while kneading, in the manner described in the second example. The material thus obtained is reduced to the form of a powder and mixed with an acetone solution containing 50 grams of urea formaldehyde soluble resin, which is commercially available under the registered trade mark "Plastopal H." After volatilization of the solvent, the material is squeezed into the desired shape at ordinary temperatures, it being possible to apply a pressure of 5000 kg./cm.^{2}.

Example IV

1 kg. of magnetic powder is intimately mixed with about 100 cm.^{3} of a 2 normal solution of aqua-ammonia containing 30 milliequivalents of cupri-ion and 45 milliequivalents of phosphate ion per 100 cm.^{3} and the mixture is evaporated to dryness and powdered, while kneading, in the manner described in the second example. The powder is agitated in a solution containing 60 grams of a hardening phenolformaldehyde condensation product and is then evaporated to dryness while shaking. The material thus obtained is reduced to the form of a powder and then squeezed at ordinary temperatures at a pressure of about 5000 kg./cm.^{2}. The cores thus obtained are hardened in a furnace by heating for three hours at 130° C.

The quantities, indicated in the examples described, of the substances used are chosen to accord with the specific weight of the iron and an average size of the particles of 3μ as possessed by certain known commercial powdered iron by means of which the invention yields particularly favorable results.

We claim:

1. The steps in a process of providing particles of ferro-magnetic material with a coating of insulation material substantially solely by precipitation on the outer surface of the particles, of an insulation material derived from a chemical compound and without causing any substantial reaction between the particles and the chemical compound, which comprise intimately mixing comminuted ferro-magnetic material with a compound comprising a 2 normal solution of aqua-ammonia containing zinc ion and phosphate ion, and drying the mixture while subjecting it to substantially continuous agitation whereby zinc phosphate is caused to be precipitated on the outer surface of the particles.

2. The process described in claim 1 characterized by that the drying of the mixture is accomplished by applying heat thereto.

3. The steps in the method of providing particles of ferro-magnetic material with a coating of insulation material substantially solely by precipitation on the outer surface of the particles of an insulation material derived from a chemical compound and without causing any substantial reaction between the particles and the chemical compound, which comprise intimately mixing in the proportion of about one kilogram of comminuted ferro-magnetic material with a compound comprising one litre of 2 normal solution of aqua-ammonia containing sixty milliequivalents of zinc ion and sixty milliequivalents of phosphate ion, drying the mixture while subjecting it to substantially continuous agitation to thereby precipitate zinc phosphate on the surface of the magnetic particles.

4. The process described in claim 1 characterized by that the mixture is made by mixing with each kilogram of magnetic material approximately one hundred cubic centimeters of the two normal solution of aqua-ammonia containing thirty milliequivalents of the zinc ion and forty-five milliequivalents of the phosphate ion whereby the mixture is in the form of a paste and by that the drying of the resulting mixture is accomplished by heating the same to a comparatively low temperature and kneading it to provide for proper mixing thereof.

5. The steps in a process of providing particles of ferro-magnetic material with a coating of insulation material substantially solely by precipitation on the outer surface of the particles of an insulation material derived from a chemical compound and without causing any substantial reaction between the particles and the chemical compound, which comprise intimately mixing comminuted magnetic material with a phosphoric acid metal compound comprising aqua-ammonia phosphate-ion and one of the elements of the group consisting of zinc-ion and cupri-ion, and treating the resulting mixture so as to form around each of said particles a thin layer of said compound.

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