

- [54] **METHOD OF MAKING STEEL POWDER PARTICLES OF SELECT ELECTRICAL RESISTIVITY**
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- [51] Int. Cl.**B22f 9/00**
- [58] Field of Search.....264/111, 104

[56] **References Cited**

UNITED STATES PATENTS

3,166,615	1/1965	Farrell.....	264/111
3,216,074	11/1965	Harrison.....	264/111
2,979,401	4/1961	Szymaszek	264/111

3,414,641 12/1968 Miller.....264/104

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[57] **ABSTRACT**

A method of making a compressed steel powder article having high resistivity to be used as an electrical component. Steel powder having a particle size finer than 28 mesh is treated with a non-aqueous solution of an organic thermosetting resin to coat the particles with the resin. After evaporation of the solvent and curing of the resin, the coated particles are compressed to form a non-sintered, compressed steel powder article having high electrical resistivity. The compressed article can be subsequently heated to an elevated temperature to stress relieve the article and stabilize the resistivity.

5 Claims, No Drawings

METHOD OF MAKING STEEL POWDER PARTICLES OF SELECT ELECTRICAL RESISTIVITY

BACKGROUND OF THE INVENTION

This invention relates to a method of making a compressed steel powder article having a select electrical resistivity.

Rotors, stators, and other magnetic cores, for use in electrical equipment are usually not formed of single piece wrought materials due to high eddy current losses developed in the low resistivity wrought iron. The conventional practice in forming rotors, stators and magnetic cores is to use a laminated structure formed from a stack of thin rolled ferrous sheets. More recently, compressed steel powder has been utilized to form high resistivity electrical components by a process in which the steel particles are initially coated with an electrically insulate coating prior to compressing into the desired shape. U.S. Pat. No. 3,245,841 describes a process of producing high electrical resistivity steel powder by treating the steel powder with a phosphoric acid and chromic acid aqueous solution to provide a surface coating on the steel particles, consisting principally of iron phosphate and chromium compounds.

It has been found that on a commercial scale the water used in the treating solution, such as that disclosed in the aforementioned patent, cannot be evaporated from a large mass of steel powder without oxidation of the steel due to the difficulty in aerating the powder mass. Oxidation of the steel particles results in a loss of mechanical properties in the compressed part and lowers the density of the compressed part.

It has also been found that when preparing electrical components from steel powder that some compressed parts should be stress relieved in order to obtain stability of resistivity under operating conditions, which can be up to 300°F, although this may not be necessary in all applications. Thus, any electrically insulating coating applied to the particles must be capable of withstanding stress relieving without decomposing or reacting with the steel, if the part is to be subjected to a stress relieving treatment.

SUMMARY OF THE INVENTION

The invention is directed to a method of making a compressed steel powder article having a select electrical resistivity which can be used in a wide variety of electrical components. In accordance with the invention, steel powder having a particle size finer than about 28 mesh is initially treated with a non-aqueous solution of an uncured organic thermosetting resin to coat the individual particles of steel powder. After evaporation of the solvent, the coated particles are compressed to form a non-sintered compressed powder article. In the compressed part, the cured resin should be capable of maintaining its high electrical resistivity without decomposing or reacting with the base metal at temperatures above 250°F and up to 1,200°F. To stabilize the resistivity of the article at operation conditions, the compressed article can be stress relieved at temperatures up to 1,200°F.

The resulting heat treated, compressed steel powder part has a high stabilized resistivity and can be operated at elevated temperatures without substantial variation in resistivity.

As the coating is applied to the steel particles by use of a non-aqueous solution, oxidation of the individual steel particles will be prevented during the treating operation. This insures that the mechanical properties of the compressed part will be maintained and provides improved uniformity from part-to-part.

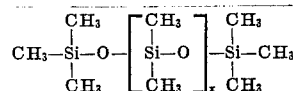
DESCRIPTION OF THE PREFERRED EMBODIMENT

The steel powder to be employed can be prepared by the method shown in the U.S. Pat. No. 3,325,277 to Huseby, in which a stream of molten steel is atomized by a sheet of high pressure water to form irregularly shaped particles which are subsequently heat treated to soften the particles and reduce the carbon and residual oxygen content.

The steel powder has a carbon content generally in the range of 0.001 to 0.1 percent and preferably less than 0.05 percent. In addition, the steel may contain alloying additions such as nickel, silicon aluminum, and the like, up to about 5 percent by weight, depending upon the magnetic characteristics desired.

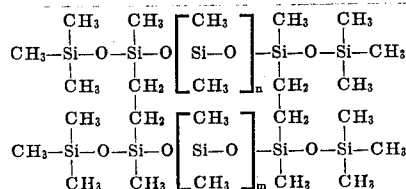
The size of the steel powder is not particularly critical and the powder generally has a size finer than about 28 mesh (590 microns). There is no limit to the fineness of the steel powder, but from a practical standpoint, if the particle size is finer than 100 mesh, the cost of the powder and the mechanical strength of the article are diminished.

The steel particles are individually coated with an uncured organic thermosetting resin. Silicone resins have proven very satisfactory as the coating material. Polyorgano-siloxanes are characterized by a molecular backbone of alternate atoms of silicon and oxygen with organic groups attached to the silicon atoms. The type of organic groups and the extent of crosslinking determine whether the silicone will be fluid, elastomeric or resinous. The basic polydimethylsiloxane structure is as follows:



where X is an integer of at least 5.

More specifically, the following silicone resin can be employed:



where both n and m are integers having a value in the range of 200 to 500.

In addition, polyimide resins of the type described in U.S. Pat. Nos. 3,179,614 and 3,179,634 can also be utilized as the coating material.

The amount of the resin to be used is important in carrying out the invention, and generally, the resin is employed in an amount from 0.06 to 1.0 percent by

weight of the steel powder, with approximately 0.25 to 0.50 percent being preferred.

The resin is applied in the uncured state to the individual dual steel particles by means of a non-aqueous treating solution. The solvent to be employed in the solution can be any volatile organic solvent, capable of dissolving the resin to be used. In general, solvents such as toluene, xylene, benzene, and the like, may be employed. As the solvent is subsequently evaporated and is not present in the final product, the particular nature of the solvent is not critical and any volatile readily available organic solvent may be employed.

The concentration of the treating solution is not critical and the solution can have any degree of dilution which will facilitate mixing with the steel powder. It has been found that a concentration in which 1 gram of the resin is dissolved in 160 to 240 ml of the solvent is satisfactory.

It is contemplated that a finely divided inert filler can be incorporated with the resin. The filler should have a substantially smaller particle size than the steel powder, and preferably has a particle size finer than 10 microns. The filler may take the form of quartz, diamotaceous earth, kaolin, talc, aluminum silicate, calcium carbonate, and the like. The amount of filler, if used, is not critical and the resin may contain from 0.01 to 3 parts by weight of filler to one part resin. When a filler is utilized, the combined weight of the filler and resin should be from 0.06 to 1.0 percent by weight of the steel powder.

As the filler will not be dissolved by the solvent, the particles of filler will be dispersed or suspended in the treating solution. In this case, when the treating solution is mixed with steel powder, the resin and the filler will coat the steel particles.

The steel powder and the treating solution can be mixed together by use of conventional blending equipment such as a rotating drum. It is not necessary to heat the materials and the materials are generally mixed together at room temperature. During the mixing operation, the treating solution will tend to completely coat or encapsulate the individual steel particles.

After the mixing operation, the solvent is evaporated. While heating is not necessary for evaporation of the solvent, under normal conditions, heating is utilized in order to accelerate the evaporation. Following evaporation of the solvent, the resin is cured. While the resin will crosslink or cure at room temperature, heat is preferred to accelerate the cure. In practice, it has been found that heating the coated particles to a temperature in the range of about 150° to 250°F for a period of 15 to 60 minutes will not only evaporate the solvent during the initial stage of the heating treatment, but will also cure the resin in the latter state of this heat treatment.

The thickness of the cured coating is not critical, but it is believed that the coating, in most cases, is continuous to virtually encapsulate the individual steel particles and is generally less than 1 micron in thickness. Due to the very thin nature of the coating, the particles after curing of the resin, will not adhere together, but the powder mass will still be flowable although the rate of flow will not be as great as the uncoated particles.

After curing of the coating, the coated particles are compressed into the shape of the desired component or

article. The compressing is preferably carried out at room temperature, although temperatures up to the decomposition temperature of the resin can be used. The coated powder is compressed preferably with a pressure in excess of 50 tsi and generally in the range of about 30 to 100 tsi. During the compression, the coating will flow and squeeze between the particles. As the compression is normally done without heating there will be no sintering of the steel particles and the particles will be held together primarily by the mechanical interlock between the irregular particles and the adhesive nature of the coating.

In the compressed part, the cured resin is characterized by the ability to maintain its high electrical resistance without decomposing or reacting with the steel when heated to a temperature in the range of 250° to 1,200°F

Following compression, the compressed part can be stress relieved by heating to a temperature in the range from about 500° to 1,200 °F and below the decomposition temperature of the resin coating. The heat treatment serves a multiple function in that it removes stress from the compressed part, stabilizes the resistivity so that the resistivity will be less likely to change during service, and increases the mechanical strength of the compressed part. If the compressed part is to be stress relieved, the resin to be used as the coating should have a decomposition temperature in the upper portion of the 250°F to 1,200°F so that the part can be heated to the stress relieving temperature without decomposition of the resin. If the part is not to be stress relieved, then a resin having a lower decomposition temperature can be used.

After stress relieving treatment the part is cooled to room temperature.

It has been found that improved properties are obtained if the resin is fully cured prior to compression, rather than curing the resin during the stress relieving treatment and following compression. It is believed that fully curing the resin prior to compression volatilizes all of the solvent, so that there is no residual solvent present in the particle mass which would adversely affect the compression.

The compressed part has a density of at least 90 percent of theoretical density of iron and generally the treated powder has a compressibility greater than 7.4 grams/cc at a compaction pressure of 60 tsi.

The compressed iron powder article of the method of the invention can be used for a wide variety of electrical components such as rotors, stators, transformer cores, chokes, and the like. The electrical resistivity of the compressed part can vary between 15 to 100,000 microhm cm. with the particular resistivity depending on the ultimate function and use of the electrical component.

The use of the silicone resin as the coating material has the added advantage that it serves as a lubricant during the compression step. By using the silicone resin it is not necessary to lubricate the die with a separate lubricant during the compression step.

The following examples illustrate the process of the invention.

EXAMPLE I

908 grams of steel powder having a carbon content of 0.005 percent and having a particle size in the range of 28 to 100 mesh were mixed with 50 ml of a 0.25 percent by weight toluene solution of silicone resin GE-RTV 102. After thorough mixing of the ingredients the resulting coated powder was heated in a circulating oven at a temperature of 250°F to evaporate the toluene and cure the resin.

Following the heating the coated particles were compressed at room temperature at 60 tsi into a part having the dimensions $1\frac{1}{4} \times \frac{1}{2} \times \frac{1}{4}$ inches. Subsequently the compressed part was heated to a temperature of 800°F for a period of 1 hour to stress relieve the part and stabilize the resistivity.

The resulting heat treated steel powder part had a density of 7.41 grams/cc. The part had a resistivity of 15,000 microhm cm., as pressed, and a resistivity of 1,800 microhm cm. after stress relieving.

The compressed part had a transverse rupture strength of 4,830 psi, as pressed, and 8,590 psi after the stress relieving treatment.

EXAMPLE II

908 grams of steel powder having a carbon content of 0.005 percent and a particle size finer than 28 mesh were mixed with 170 ml of a 0.25 percent toluene solution of silicone resin GE RTV-106.

After thorough mixing of the ingredients to coat the particles the mixture was heated in a circulating oven to a temperature of 250°F to evaporate the solvent and cure the resin. The coated particles were then compressed at room temperature and at a pressure of 60 tsi into an article having the dimensions $1\frac{1}{4} \times \frac{1}{2} \times \frac{1}{4}$ inches.

The compressed part was then stress relieved at a temperature of 800°F for a period of 1 hour.

The resulting stress relieved part had a density of 7.40 grams/cc, and a resistivity, as pressed, of 7,300 microhm cm. and a resistivity, after stress relieving, of 1,300 microhm cm.

EXAMPLE III

A compressed steel powder part was prepared in a manner similar to that of Example I, except the treating solution contained 0.06 percent of silicone resin GE RTV-102.

The resulting compressed and stress relieved part had a density of 7.43 grams/cc and the compressed powder part had a resistivity of 16,500 microhm cm., as pressed, and a resistivity of 1,200 microhm cm. after stress relieving.

The compressed article had a transverse rupture strength of 5,800 psi, as pressed, and 10,000 psi after stress relieving.

Various modes of carrying out the invention are contemplated as being within the scope of the following claims particularly pointing out and distinctly claiming the subject matter which is regarded as the invention.

I claim:

1. A method of preparing a compressed steel powder article having a predetermined electrical resistivity to be used in electrical apparatus, comprising the steps of:

- a. dissolving an uncured thermosetting resin in an organic non-aqueous solvent to provide a treating solution,
- b. blending the solution with a quantity of steel particles having a particle size finer than 28 mesh and a carbon content in the range of 0.001 to 0.10 percent by weight to coat said particles, said resin on a solids basis being present in an amount of 0.06 to 1.0 percent by weight of the steel particles,
- c. evaporating the solvent,
- d. curing the resin to provide a flowable mass of coated particles, and
- e. compressing the coated particles at a pressure in the range of 30 to 100 tsi to mechanically interlock the steel particles and form a compressed steel article having a density greater than 90 percent of the theoretical density of pure iron and having an electrical resistivity in the range of 15 to 100,000 microhm cm.

2. The method of claim 1, wherein the compressing is done at a temperature below the sintering temperature of the steel particles.

3. The method of claim 1, wherein the compressing is done at room temperature.

4. A method of preparing a compressed steel powder article having a predetermined electrical resistivity to be used in an electrical apparatus comprising the steps of:

- a. dissolving an uncured thermosetting resin having a decomposition temperature in the range of 500° to 1,200°F in an organic non-aqueous solvent to provide a treating solution,
- b. blending the solution with a quantity of steel particles having a particle size finer than 28 mesh and a carbon content in the range of 0.001 to 0.10 percent by weight to coat said particles, said resin on a solids basis being present in an amount of 0.06 to 1.0 percent by weight of the steel particles,
- c. evaporating the solvent,
- d. curing said resin to provide a flowable mass of coated particles,
- e. compressing the coated particles at a pressure in the range of 30 to 100 tsi to mechanically interlock the steel particles and form a compressed steel article having a density greater than 90 percent of the theoretical density of pure iron and having an electrical resistivity in the range of 15 to 100,000 microhm cm., and
- f. heating the compressed article to a temperature in the range of 500° to 1,200°F and below the decomposition temperature of the resin to stress relieve the article and stabilize the resistivity.

5. The method of claim 4, wherein said resin is selected from the group consisting of silicone resins and polyimide resins.

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