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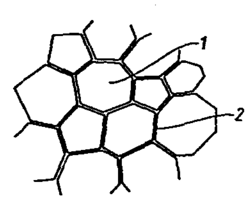
(54) **Method for manufacturing an object of soft-magnetic material by bonding together a mass of powder grains.**

(57) A method for manufacturing an object of soft-magnetic material by bonding together a mass of powder grains of iron-based soft-magnetic material in the form of pure iron or in the form of iron containing silicon, aluminium and/or titanium. The grains are provided either with a coating (2) containing iron oxide chemically bonded to the grains and, at least in those cases where the iron-based material consists of pure iron, containing in addition thereto an oxide of silicon, aluminium and/or titanium, or the grains are provided with a coating (2) containing a reaction product of iron oxide, which oxide is chemically bonded to the grains, and an oxide of silicon, aluminium and/or titanium. The grains provided with said coating are subjected to an isostatic pressing at a temperature of at most 900°C and at a pressure necessary for forming a coherent, compact body of the powder mass. The coating preferably has a thickness of 0.01 - 0.5 µm.



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Method for Manufacturing an Object of Soft-Magnetic Material by Bonding Together a Mass of Powder Grains

The invention relates to a method for manufacturing an object of soft-magnetic material by bonding together a mass of powder grains according to the introductory part of claim 1.

Proposals have been made for replacing magnetic cores of laminated sheet metal for stators in electrical machines by magnetic cores manufactured from powder. The latter magnetic cores are manufactured from powder the grains of which consist of a soft-magnetic material with a high saturation magnetization, such as pure iron or silicon-containing iron, for example iron containing 3 % silicon, by bonding together the powder grains by a resinous binder under the application of heat and pressure.

It is known that powder cores for high frequency applications can be manufactured by providing powder grains of a soft-magnetic material with a coating of a ceramic binder and by then pressing the powder grains into an object which is subsequently subjected to a heat treatment.

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When pressing magnetic cores of powder grains intended to replace magnetic cores of laminated sheet metal, problems arise in obtaining - at the same time - the necessary property values with respect to eddy current losses, magnetizability, hysteresis losses and mechanical strength.

The invention aims at developing a method of the above mentioned kind which allows to manufacture objects from powder of iron-based soft-magnetic material, which combine high magnetizability and high mechanical strength with low eddy current losses and low hysteresis losses.

In order to achieve this aim the invention suggests a method according to the preamble of claim 1 which is characterized by the features of the characterizing part of claim 1.

Further particulars of the method according to the invention are characterized by the features of the subsequent claims.

A feasible explanation of the results obtained according to the invention may be the following: Through the chemical bond of the iron oxide or the reaction product of the iron oxide and other oxides to the powder grain material, the coating on the grains is well anchored to said powder grain material, which contributes to a good mechanical strength of the finished object. Because the coating is at least relatively resistent to deformations at higher temperatures but still has a certain flexibility in thin layers so that it can be adapted to changes of the shape of the grains of the iron-bases material during pressing, and because the pressing is carried out as an isostatic pressing, the coating can be brought to form, in the finished object, thin layers of uniform thickness thus giving the object a compact structure. The electrically insulating character of the layers results in both low eddy current losses and good

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magnetizability of the finished object. Further, owing to the fact that the pressing can be carried out at relatively high temperatures, internal stresses which may occur in the grain material may be removed, which results in low hysteresis losses of the finished object. If, however, the temperature during the pressing becomes too high, such as above 900°C, no reduction is obtained, in any case no considerable reduction, of the eddy current losses compared with those of solid iron-based material of the same kind, which is probably due to metallic contacts then easily arising between the grains.

The grains of the iron-based material may consist of at least approximately pure iron, preferably containing at least 99.95% Fe. Here, and in the following, the symbol "%" relates to percentage by weight. The grains may also consist of iron containing silicon, aluminium, and/or titanium. The content of silicon may suitably amount to 0.01-8 %, the content of aluminium to 0.001 - 2% and the content of titanium to 0.001 - 1%. Regardless of the type of iron-based material, the carbon content should be below 0.005%.

At least the main part of the grains of the iron-based material suitably has a size of 50-1000 μm and the coating suitably a thickness of 0.01 - 0.5 μm. The layers formed by the coatings between two adjacent grains have, on an average, a thickness of 0.02 - 1 μm. The ratio between the average thickness of said layers and the average grain size of the iron-based material is preferably between the limits 10^{-4} to 10^{-2}.

The iron oxide on the grains can advantageously be accomplished by oxidation of the powder grains in a fluidized bed reactor with water vapour or air at a temperature of around 400°C. If the iron-based material consists of pure iron, at least an oxide of silicon, aluminium or titanium is supplied to the surface of the oxidized grains to form the coating. This can be performed from a colloidal solution of the oxide in question with water. The oxide can also be supplied otherwise. The silicon oxide, for example, may be supplied by coating the oxidized grains with a hydrolyzable organic silicon compound, such as tetraethyl silicate, which is then hydrolyzed while forming a silicon dioxide. The amount of supplied oxide shall be sufficient to form - with the iron oxide during a subsequent heat treatment, suitably at a temperature of above 800°C in inert atmosphere, either in connection with the isostatic pressing or in a separate stage prior thereto - a silicate, aluminate and/or a titanate, which is active as a binder in the pressed object. Also if the iron-based material consists of iron containing silicon, aluminium or iron, the described method of supplying oxides of silicon, aluminium and/or titanium from outside can be employed. If the iron-based material is of the latter kind, however, the silicon, aluminium and/or titanium included in the iron-based material can be utilized to form the silicate, aluminate and/or titanate with the iron oxide formed during the previously mentioned oxidation of the grains. The formation of silicate, aluminate and/or titanate takes place at elevated temperature, suitably at above 800°C, and suitably in an atmosphere of moist hydrogen gas. During said heat treatment, silicon, aluminium and/or titanium in the iron-based material reacts with the iron oxide present at the surface of the grains to form silicon dioxide, aluminium trioxide and/or titanium dioxide, whereby the iron oxide is reduced to iron. The primarily formed oxide of the kind mentioned further reacts with iron oxide remaining at the surface of the grains thereby forming silicate, aluminate and/or titanate. This can take place either in a separate stage prior to the isostatic pressing or in conjunction with the isostatic pressing. If the content of silicon, aluminium and/or titanium in the ironbased material is sufficient to form a sufficient amount of silicate, aluminate and/or titanate, no oxide of these substances need be supplied from outside. If the content is insufficient, a supplementary amount of oxide may be subsequently supplied, such as described in connection with the supply of such oxide to oxidized grains of pure iron, where this supply is necessary.

The isostatic pressing is suitably carried out at a temperature of 600-900°C, preferably at a temperature of 600-775°C, and suitably at a pressure of 10 - 200 MPa, preferably at a pressure of at least 50 MPa.

The invention will now be explained in greater detail by describing some examples with reference to the accompanying figure, which schematically shows the structure of the material in an object manufactured according the the invention.

The figure shows the structure of part of an object built up of grains 1 of an iron-based soft-magnetic material, which are bonded to each other by means of a binder 2 formed of coatings on the individual grains. Examples of such coatings are described in the following:

Example 1

Substantially spherical grains of approximately pure iron containing 99,99% Fe and less than 0.005% C and having a size of 45-200 μm are oxidized at a temperature of 400°C in water vapour so that the grains receive a coating of iron oxide having a thickness of about 0.01 μm, which takes less than 1 minute. The oxidized grains are dipped into a colloidal solution of silicon dioxide in water and are then dried, thus receiving a coating of silicon dioxide having a thickness of 0.04 μm. The grains are then placed in a capsule of steel sheet having low carbon content and having the same shape as, but larger dimensions than, the object which is to be manufactured. The capsule with its contents is evacuated and sealed. It is thereafter placed in a high pressure furnace where it is heated to 700°C before being subjected to a pressure of 100 MPa by an inert gas such as argon. The temperature is then raised to 750°C while maintaining the pressure. When the grain material has been subjected to the pressure at this temperature for two hours, the capsule with its contents is allowed to cool and the capsule is removed. Thereby the oxides in the coating react to form silicate. The object obtained during the isostatic pressing forms a coherent body with a densitiy exceeding 99 % of the theoretical density and with a good mechanical strength.

Instead of silicon dioxide there may be used aluminium oxide or titanium dioxide (preferably in the form of anatase) or a mixture of at least two of these oxides to obtain the described coating on the grain covered with iron oxide.

Example 2

Substantially spherical grains of silicon steel containing 3 % Si and less than 0.005 % C and having a grain size of 150-350 µm are oxidized in water vapour at a temperature of around 400°C so that the grains receive a coating of iron oxide having a thickness of about 0.1 µm. The oxidized grains are then treated at a temperature of around 825°C in moist nitrogen gas for a few minutes so that a silicate, formed in a manner previously described, is obtained at the surface of the grains. The grains are thereafter placed in a capsule of steel sheet with a low carbon content and having the same shape as, but larger dimensions than, the object to be manufactured. The capsule with its contents is subjected to the same treatment as the filled capsule described under Example 1. The object obtained during the isostatic pressing forms a coherent body with the silicate as a binder between the grains. It has a density exceeding 99 % of the theorectical density and a good mechanical strength.

Instead of using a steel containing 3 % Si there may be used a steel containing 0.2 % aluminium ~~or a steel con-~~

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~~taining 0.2 % aluminium~~ or a steel containing 0.01 % Ti or a steel containing at least two of said substances in said contents.

The manufactured object may, among other things, be used as a magnetic core, preferably as a stator, for an electrical machine or as a magnetic core for a magnetically operated contactor. It may also constitute part of a magnetic core, for example for a stator for an electrical machine. A magnetic core for a large stator can then be manufactured in a number of parts which are arranged adjacent to each other in order to form the magnetic core.

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Claims

1. Method for manufacturing an object of soft-magnetic material by bonding together a mass of powder grains of an iron-based soft-magnetic material in the form of at least approximately pure iron or in the form of iron containing silicon, aluminium and/or titanium, which grains are provided with a coating and are subjected to pressure and heating, characterized in that the coating of the grains (1) contains iron oxide, chemically bonded to the grains, and, at least in those cases where the iron-based material consists of at least approximately pure iron, contains in addition thereto an oxide of silicon, aluminium and/or titanium, or/and that the coating of the grains contains a reaction product of iron oxide, which oxide is chemically bonded to the grains, and an oxide of silicon, aluminium and/or titanium, and that the mass of coated grains is subjected to an isostatic pressing at a temperature of at most 900°C and at a pressure necessary for forming a coherent dense body of the mass.

2. Method according to claim 1, characterized in that the density of the compressed body exceeds 99 % of the theoretical density.

3. Method according to claim 1 or 2, characterized in that the coating has a thickness of 0.01 - 0.5 μm and the grains have a size of 50-1000 μm.

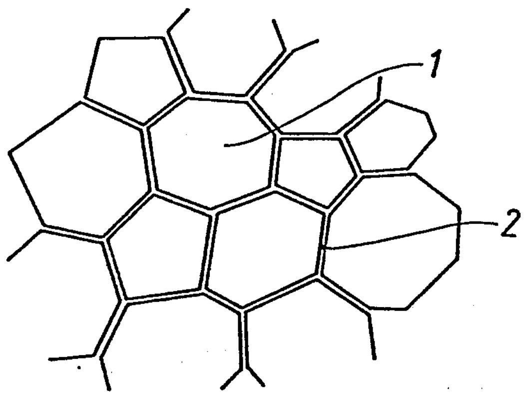
4. Method according to any of claim 1 - 3, characterized in that the iron-based material consists of at least approximately pure iron, and that the grains are provided with a coating consisting of a layer of iron oxide, formed on the grains by treating them in an oxidizing

atmosphere, and of a layer of an oxide of silicon, aluminium, and/or titanium applied on the layer of iron oxide.

5. Method according to any of claim 1- 3, characterized in that the iron-based material consists of iron containing silicon, aluminium and/or titanium, and that the grains are provided with a coating consisting of a reaction product of iron oxide, formed on the grains by treating them in an oxidizing atomosphere, and an oxide of silicon, aluminium and/or titanium included in the iron-based material.

6. Method according to any of claim 1 - 5, characterized in that the isostatic pressing is carried out at a temperature of 600-900°C and at a pressure of at least 10 MPa.

7. Method according to any of claim 1 - 5, characterized in that the isostatic pressing is carried out at a temperature of 600-775°C and at a pressure of at least 50 MPa.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