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(54) **New composition and method**

(57) The present invention concerns a composite iron- based powder mix suitable for soft magnetic applications such as inductor cores. The present invention

also concerns a method for producing a soft magnetic component and the component produced by the method.

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DescriptionField of the invention

5 **[0001]** The present invention concerns a soft magnetic composite powder material for the preparation of soft magnetic components as well as the soft magnetic components which are obtained by using this soft magnetic composite powder. Specifically the invention concerns such powders for the preparation of soft magnetic components materials working at high frequencies, the components suitable as inductors or reactors for power electronics.

10 Background of the invention

[0002] Soft magnetic materials are used for various applications, such as core materials in inductors, stators and rotors for electrical machines, actuators, sensors and transformer cores. Traditionally, soft magnetic cores, such as rotors and stators in electric machines, are made of stacked steel laminates. Soft magnetic composites may be based on soft magnetic particles, usually iron-based, with an electrically insulating coating on each particle. By compacting the insulated particles optionally together with lubricants and/or binders using the traditionally powder metallurgy process, soft magnetic components may be obtained. By using the powder metallurgical technique it is possible to produce such components with a higher degree of freedom in the design, than by using the steel laminates as the components can carry a three dimensional magnetic flux and as three dimensional shapes can be obtained by the compaction process.

15 **[0003]** The present invention relates to an iron-based soft magnetic composite powder, the core particles thereof being coated with a carefully selected coating rendering the material properties suitable for production of inductors through compaction of the powder followed by a heat treating process.

[0004] An inductor or reactor is a passive electrical component that can store energy in form of a magnetic field created by the electric current passing through said component. An inductor's ability to store energy, inductance (L) is measured in henries (H). Typically an inductor is an insulated wire wound as a coil. An electric current flowing through the turns of the coil will create a magnetic field around the coil, the field strength being proportional to the current and the turns/length unit of the coil. A varying current will create a varying magnetic field which will induce a voltage opposing the change of current that created it.

25 **[0005]** The electromagnetic force (EMF) which opposes the change in current is measured in volts (V) and is related to the inductance according to the formula;

$$v(t) = L \, di(t)/dt$$

30 (L is inductance, t is time, v(t) is the time-varying voltage across the inductor and i(t) is the time-varying current.)

[0006] That is; an inductor having an inductance of 1 henry produces an EMF of 1 volt when the current through the inductor changes with 1 ampere/second.

35 **[0007]** Ferromagnetic- or iron- core inductors use a magnetic core made of a ferromagnetic or ferrimagnetic material such as iron or ferrite to increase the inductance of a coil by several thousand by increasing the magnetic field, due to the higher permeability of the core material.

[0008] The magnetic permeability, ρ , of a material is an indication of its ability to carry a magnetic flux or its ability to become magnetised. Permeability is defined as the ratio of the induced magnetic flux, denoted B and measured in newton/ampere*meter or in volt*second/meter², to the magnetising force or field intensity, denoted H and measured in amperes/meter, A/m. Hence magnetic permeability has the dimension volt*second/ampere*meter. Normally magnetic permeability is expressed as the relative permeability $\mu_r = \rho / \mu_0$, relative to the permeability of the free space, $\mu_0 = 4 * \pi * 10^{-7} \text{Vs/Am}$.

40 **[0009]** Permeability may also be expressed as the inductance per unit length, henries/meter. Magnetic permeability does not only depend on material carrying the magnetic flux but also on the applied electric field and the frequency thereof. In technical systems it is often referred to the maximum relative permeability which is maximum relative permeability measured during one cycle of the varying electrical field.

[0010] An inductor core may be used in power electronic systems for filtering unwanted signals such as various harmonics. In order to function efficiently an inductor core for such application shall have a low maximum relative permeability which implies that the relative permeability will have a more linear characteristic relative to the applied electric field, i.e. stable incremental permeability, μ_{Δ} (as defined according to $\Delta B = \mu_{\Delta} * \Delta H$), and high saturation flux density. This enables the inductor to work more efficiently in a wider range of electric current, this may also be expressed as that the inductor has "good DC-bias". DC-bias may be expressed in terms of percentage of maximum incremental permeability

at a specified applied electrical field, e.g. at 4 000 A/m. Further low maximum relative permeability and stable incremental permeability combined with high saturation flux density enables the inductor to carry a higher electrical current which is *inter alia* beneficial when size is a limiting factor, a smaller inductor can thus be used.

5 [0011] One important parameter in order to improve the performance of soft magnetic component is to reduce its core loss characteristics. When a magnetic material is exposed to a varying field, energy losses occur due to both hysteresis losses and eddy current losses. The hysteresis loss is proportional to the frequency of the alternating magnetic fields, whereas the eddy current loss is proportional to the square of the frequency. Thus at high frequencies the eddy current loss matters mostly and it is especially required to reduce the eddy current loss and still maintaining a low level of hysteresis losses. This implies that it is desired to increase the resistivity of magnetic cores.

10 [0012] In the search for ways of improving the resistivity different methods have been used and proposed. One method is based on providing electrically insulating coatings or films on the powder particles before these particles are subjected to compaction. Thus there are a large number of patent publications which teach different types of electrically insulating coatings. Examples of published patents concerning inorganic coatings are the U.S. Pat. No. 6,309,748, U.S. Pat. No. 6,348,265 and U.S. No. 6,562,458. Coatings of organic materials are known from e.g. the U.S. Pat. No. 5,595,609. 15 Coatings comprising both inorganic and organic material are known from e.g. the U.S. Pat. Nos. 6,372,348 and 5,063,011 and the DE patent publication 3,439,397, according to which publication the particles are surrounded by an iron phosphate layer and a thermoplastic material. European Patent EP1246209B1 describes a ferromagnetic metal based powder wherein the surface of the metal-based powder is coated with a coating consisting of silicone resin and fine particles of clay minerals having layered structure such as bentonite or talc.

20 [0013] US6,756,118B2 reveals a soft magnetic powder metal composite comprising a least two oxides encapsulating powdered metal particles, the at least two oxides forming at least one common phase.

[0014] The patent application JP2002170707A describes an alloyed iron particle coated with a phosphorous containing layer, the alloying elements may be silicon, nickel or aluminium. In a second step the coated powder is mixed with a water solution of sodium silicate followed by drying. Dust cores are produced by moulding the powder and heat treat 25 the moulded part in a temperature of 500-1000°C.

[0015] Sodium silicate is mentioned in JP51-089198 as a binding agent for iron powder particles when producing dust cores by moulding of iron powder followed by heat treating of the moulded part.

[0016] In order to obtain high performance soft magnetic composite components it must also be possible to subject the electrically insulated powder to compression moulding at high pressures as it is often desired to obtain parts having 30 high density. High densities normally improve the magnetic properties. Specifically high densities are needed in order to keep the hysteresis losses at a low level and to obtain high saturation flux density. Additionally, the electrical insulation must withstand the compaction pressures needed without being damaged when the compacted part is ejected from the die. This in turn means that the ejection forces must not be too high.

[0017] Furthermore, in order to reduce the hysteresis losses, stress releasing heat treatment of the compacted part is required. In order to obtain an effective stress release the heat treatment should preferably be performed at a tem- 35 perature above 300°C and below a temperature where the insulating coating will be damaged, in an atmosphere of for example nitrogen, argon or air, or in vacuum.

[0018] The present invention has been done in view of the need for powder cores which are primarily intended for use at higher frequencies, i.e. frequencies above 2 kHz and particularly between 5 and 100 kHz, where higher resistivity 40 and lower core losses are essential. Preferably the saturation flux density shall be high enough for core downsizing. Additionally it should be possible to produce the cores without having to compact the metal powder using die wall lubrication and/or elevated temperatures. Preferably these steps should be eliminated.

[0019] In contrast to many used and proposed methods, in which low core losses are desired, it is an especial advantage of the present invention that it is not necessary to use any organic binding agent in the powder composition, which 45 powder composition is later compacted in the compaction step. Heat treatment of the green compact can therefore be performed at higher temperature without the risk that any organic binding agent decomposes; a higher heat treatment temperature will also improve the flux density and decrease core losses. The absence of organic material in the final, heat treated core also allows the core to be used in environments with elevated temperatures without risking decreased strength due to softening and decomposition of an organic binder, and improved temperature stability is thus achieved.

50 Objects of the invention

[0020] An object of the invention is to provide a new iron- based composite powder comprising a core of an iron based powder the surface thereof coated with a new composite electrical insulated coating. The new iron based composite 55 powder being especially suited to be used for production of inductor cores for power electronics.

[0021] Another object of the invention is to provide a method for producing such inductor cores.

[0022] Still another object of the invention is to provide an inductor core having "good" DC-bias, low core losses and high saturation flux density.

[0023] The present invention provides an iron powder mixture and process methods for treating said mixture which can be used to prepare e.g. inductors having high saturation flux density, lower core loss, and the manufacturing process thereof can be simplified.

5 Summary of the invention

[0024] At least one of these objects is accomplished by:

- 10 - A coated iron-based powder composition, the coating comprising a first phosphorous containing layer and a second layer containing a combination of alkaline silicate and particles of clays containing defined phyllosilicates, wherein the iron-based powder composition comprises a mixture of iron-powder and sendust. According to an embodiment the coating is constituted of the above two layers alone.
- 15 - A method for producing a inductor core comprising the steps of:
 - a) providing a coated iron powder composition as above,
 - b) compacting the coated iron and sendust powder mixture, optionally mixed with a lubricant, in a uniaxial press movement in a die at a compaction pressure between 400 and 1200 MPa
 - 20 c) ejecting the compacted component from the die.
 - d) heat treating the ejected component at a temperature up to 800°C.
- A component, such as an inductor core, produced according to above.

25 Detailed description of the invention

[0025] The composition may be a composite iron-based powder composition comprising core particles coated with a layer containing an alkaline silicate combined with a clay mineral containing a phyllosilicate, wherein the combined silicon- oxygen tetrahedral layer and hydroxide octahedral layers thereof preferably are electrically neutral, wherein the core particles is a mixture of

- 30 (a) iron alloy particles consisting essentially of 7% to 13% by weight silicon, 4% to 7% by weight aluminium and the balance being iron, and
- (b) atomized iron particles.

35 **[0026]** The iron alloy particles may also be referred to as "sendust" or "sendust particles".

[0027] In one embodiment, the sendust particles are coated with a phosphorous containing layer prior to coating with said alkaline silicate combined with a clay mineral containing a phyllosilicate. For brevity, this coating may be termed "alkaline silicate-coating", or "clay-coating". This coating may be e.g. kaolin- or talc-based.

40 **[0028]** In another embodiment, both the iron alloy particles and the atomized particles are coated with a phosphorous containing layer prior to coating with said alkaline silicate coating.

[0029] Throughout the text, the terms "layer" and "coating" may be used interchangeably.

[0030] The iron particles may be in the form of a pure iron powder having low content of contaminants such as carbon or oxygen. The iron content is preferably above 99.0% by weight, however it may also be possible to utilise iron- powder alloyed with for example silicon. For a pure iron powder, or for an iron- based powder alloyed with intentionally added alloying elements, the powders contain besides iron and possible present alloying elements, trace elements resulting from inevitable impurities caused by the method of production. Trace elements are present in such a small amount that they do not (or only marginally) influence the properties of the material. Examples of trace elements may be carbon up to 0.1 %, oxygen up to 0.3%, sulphur and phosphorous up to 0.3 % each and manganese up to 0.3%.

50 **[0031]** The particle size of the iron- based powder is determined by the intended use, i.e. which frequency the component is suited for. The mean particle size of the iron-based powder, which is also the mean size of the coated powder as the coating is very thin, may be between 20 to 300 μm . Examples of mean particle sizes for suitable iron-based powders are e.g. 20-80 μm , a so called 200 mesh powder, 70-130 μm , a 100 mesh powder, or 130-250 μm , a 40 mesh powder.

[0032] The iron alloy particles may consist essentially of 7% to 13% by weight silicon, 4% to 7% by weight aluminium, the balance being iron, the remainder being impurities.

55 **[0033]** Such a powder is known in the field as sendust. Typically, sendust essentially contains 84-86%Fe, 9-10%Si and 5-6%Al, on a weight basis.

[0034] The iron particles may be water atomized or gas atomized. Methods for atomizing iron are known in the literature.

[0035] The phosphorous containing coating which is normally applied to the bare iron-based powder may be applied

according to the methods described in US patent 6,348,265. This means that the iron or iron- based powder is mixed with phosphoric acid dissolved in a solvent such as acetone followed by drying in order to obtain a thin phosphorous and oxygen containing coating on the powder. The amount of added solution depends inter alia on the particle size of the powder; however the amount shall be sufficient in order to obtain a coating having a thickness between 20 and 300 nm.

[0036] Alternatively, it would be possible to add a thin phosphorous containing coating by mixing an iron-based powder with a solution of ammonium phosphate dissolved in water or using other combinations of phosphorous containing substances and other solvents. The resulting phosphorous containing coating cause an increase in the phosphorous content of the iron-based powder of between 0.01 to 0.15%.

[0037] The alkaline silicate coating is applied to the phosphorous coated iron-based powder by mixing the powder with particles of a clay or a mixture of clays containing defined phyllosilicate and a water soluble alkaline silicate, commonly known as water glass, followed by a drying step at a temperature between 20-250°C or in vacuum. Phyllosilicates constitutes the type of silicates where the silicontetrahedrons are connected with each other in the form of layers having the formula $(\text{Si}_2\text{O}_5^{2-})_n$. These layers are combined with at least one octahedral hydroxide layer forming a combined structure. The octahedral layers may for example contain either aluminium or magnesium hydroxides or a combination thereof. Silicon in the silicontetrahedral layer may be partly replaced by other atoms. These combined layered structures may be electroneutral or electrically charged, depending on which atoms are present.

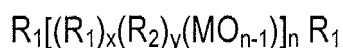
[0038] It has been noticed that the type of phyllosilicate is of vital importance in order to fulfil the objects of the present invention. Thus, the phyllosilicate shall be of the type having uncharged or electroneutral layers of the combined silicontetrahedral- and hydroxide octahedral - layer. Examples of such phyllosilicates are kaolinite present in the clay kaolin, pyrophyllit present in phyllite, or the magnesium containing mineral talc. The mean particle size of the clays containing defined phyllosilicates shall be below 15, preferably below 10, preferably below 5 μm , even more preferable below 3 μm . The amount of clay containing defined phyllosilicates to be mixed with the coated iron-based powder shall be between 0.2-5%, preferably between 0.5-4%, by weight of the coated composite iron- based powder.

[0039] The amount of alkaline silicate calculated as solid alkaline silicate to be mixed with the coated iron-based powder shall be between 0.1-0.9% by weight of the coated composite iron- based powder, preferably between 0.2-0.8% by weight of the iron-based powder. It has been shown that various types of water soluble alkaline silicates can be used, thus sodium, potassium and lithium silicate can be used. Commonly an alkaline water soluble silicate is characterised by its ratio, i.e. amount of SiO_2 divided by amount of Na_2O , K_2O or Li_2O as applicable, either as molar or weight ratio. The molar ratio of the water soluble alkaline silicate shall be 1.5-4, both end points included. If the molar ratio is below 1.5 the solution becomes too alkaline, if the molar ratio is above 4 SiO_2 will precipitate.

[0040] It may be possible to omit the second kaolin - sodium silicate coating on the Sendust particles and still achieve excellent magnetic properties. However, in order to further enhance the magnetic properties the second coating layer should cover both the Sendust and the iron powder.

[0041] In an alternative embodiment, the alkaline silicate (or clay) coating may be replaced by a metal-organic coating (second coating)

[0042] In this case, at least one metal-organic layer is located outside the first phosphorous-based layer. The metal-organic layer is of a metal-organic compound having the general formula:



wherein:

M is a central atom selected from Si, Ti, Al, or Zr;

O is oxygen;

R_1 is a hydrolysable group;

R_2 is an organic moiety and wherein at least one R_2 contains at least one amino group;

wherein n is the number of repeatable units being an integer between 1 and 20; wherein x is an integer between 0 and 1; wherein y is an integer between 1 and 2 (x may thus be 0 or 1 and y may be 1 or 2).

[0043] The metal-organic compound may be selected from the following groups: surface modifiers, coupling agents, or cross-linking agents.

[0044] R_1 in the metal-organic compound may be an alkoxy-group having less than 4, preferably less than 3 carbon atoms.

[0045] R_2 is an organic moiety, which means that the R_2 -group contains an organic part or portion. R_2 may include 1-6, preferably 1-3 carbon atoms. R_2 may further include one or more hetero atoms selected from the group consisting of N, O, S and P. The R_2 group may be linear, branched, cyclic, or aromatic.

[0046] R_2 may include one or more of the following functional groups: amine, diamine, amide, imide, epoxy, hydroxyl,

ethylene oxide, ureido, urethane, isocyanato, acrylate, glyceryl acrylate, benzyl-amino, vinyl-benzyl-amino. The R₂ group may alter between any of the mentioned functional R₂-groups and a hydrophobic alkyl group with repeatable units.

[0047] The metal-organic compound may be selected from derivatives, intermediates or oligomers of silanes, siloxanes and silsesquioxanes or the corresponding titanates, aluminates or zirconates.

[0048] According to one embodiment at least one metal-organic compound in one metal-organic layer is a monomer (n=1).

[0049] According to another embodiment at least one metal-organic compound in one metal-organic layer is an oligomer (n=2-20).

[0050] According to another embodiment the metal-organic layer located outside the first layer is of a monomer of the metal-organic compound and wherein the outermost metal-organic layer is of an oligomer of the metal-organic compound. The chemical functionality of the monomer and the oligomer is necessarily not same. The ratio by weight of the layer of the monomer of the metal-organic compound and the layer of the oligomer of the metal-organic compound may be between 1:0 and 1:2, preferably between 2:1-1:2.

[0051] If the metal-organic compound is a monomer it may be selected from the group of trialkoxy and dialkoxysilanes, titanates, aluminates, or zirconates. The monomer of the metal-organic compound may thus be selected from 3-aminopropyltrimethoxysilane, 3-aminopropyl-triethoxysilane, 3-aminopropyl-methyldiethoxysilane, N-aminoethyl-3-aminopropyl-trimethoxysilane, N-aminoethyl-3-aminopropyl-methyl-dimethoxysilane, 1,7-bis(triethoxysilyl)-4-azaheptan, triamino-functional propyl-trimethoxysilane, 3-ureidopropyl-triethoxysilane, 3-isocyanatopropyltriethoxysilane, tris(3-trimethoxysilylpropyl)-isocyanurate, 0-(propargyloxy)-N-(triethoxysilylpropyl)-urethane, 1-aminomethyl-triethoxysilane, 1-aminoethyl-methyl-dimethoxysilane, or mixtures thereof.

[0052] An oligomer of the metal-organic compound may be selected from alkoxy-terminated alkyl-alkoxy-oligomers of silanes, titanates, aluminates, or zirconates. The oligomer of the metal-organic compound may thus be selected from methoxy, ethoxy or acetoxy-terminated amino-silsesquioxanes, amino-siloxanes, oligomeric 3-aminopropyl-methoxysilane, 3-aminopropyl/propyl-alkoxy-silanes, N-aminoethyl-3-aminopropyl-alkoxy-silanes, or N-aminoethyl-3-aminopropyl/methyl-alkoxy-silanes or mixtures thereof.

[0053] The total amount of metal-organic compound may be 0.05-0.6 %, preferably 0.05-0.5 %, more preferably 0.1-0.4%, and most preferably 0.2-0.3% by weight of the composition. These kinds of metal-organic compounds may be commercially obtained from companies, such as Evonik Ind., Wacker Chemie AG, Dow Corning, etc.

[0054] The metal-organic compound has an alkaline character and may also include coupling properties i.e. a so called coupling agent which will couple to the first inorganic layer of the iron-based powder. The substance should neutralise the excess acids and acidic bi-products from the first layer. If coupling agents from the group of aminoalkyl alkoxy-silanes, -titanates, -aluminates, or -zirconates are used, the substance will hydrolyse and partly polymerise (some of the alkoxy groups will be hydrolysed with the formation of alcohol accordingly). The coupling or cross-linking properties of the metal-organic compounds is also believed to couple to the metallic or semi-metallic particulate compound which may improve the mechanical stability of the compacted composite component.

Metal or semi-metallic particulate compound

[0055] The coated soft magnetic iron-based powder may also contain at least one metallic or semi-metallic particulate compound. The metallic or semi-metallic particulate compound should be soft, having Mohs hardness less than 3.5, and constitute fine particles or colloids. The compound may preferably have an average particle size below 5 μm, preferably below 3 μm, and most preferably below 1 μm. The metallic or semi-metallic particulate compound may have a purity of more than 95%, preferably more than 98%, and most preferably more than 99% by weight. The Mohs hardness of the metallic or semi-metallic particulate compound is preferably 3 or less, more preferably 2.5 or less. SiO₂, Al₂O₃, MgO, and TiO₂ are abrasive and have a Mohs hardness well above 3.5 and is not within the scope of the invention. Abrasive compounds, even as nano-sized particles, cause irreversible damages to the electrically insulating coating giving poor ejection and worse magnetic and/or mechanical properties of the heat-treated component.

[0056] The metallic or semi-metallic particulate compound may be at least one selected from the group: lead, indium, bismuth, selenium, boron, molybdenum, manganese, tungsten, vanadium, antimony, tin, zinc, cerium.

[0057] The metallic or semi-metallic particulate compound may be an oxide, hydroxide, hydrate, carbonate, phosphate, fluorite, sulphide, sulphate, sulphite, oxychloride, or a mixture thereof.

[0058] According to a preferred embodiment the metallic or semi-metallic particulate compound is bismuth, or more preferably bismuth (III) oxide. The metallic or semi-metallic particulate compound may be mixed with a second compound selected from alkaline or alkaline earth metals, wherein the compound may be carbonates, preferably carbonates of calcium, strontium, barium, lithium, potassium or sodium.

[0059] The metallic or semi-metallic particulate compound or compound mixture may be present in an amount of 0.05-0.5 %, preferably 0.1-0.4%, and most preferably 0.15-0.3% by weight of the composition.

[0060] The metallic or semi-metallic particulate compound is adhered to at least one metal-organic layer. In one

embodiment of the invention the metallic or semi-metallic particulate compound is adhered to the outermost metal-organic layer.

[0061] The metal-organic layer may be formed by mixing the powder by stirring with different amounts of first a basic aminoalkyl-alkoxy silane (Dynasylan®/Ameo) and thereafter with an oligomer of an aminoalkyl/alkyl-alkoxy silane (Dynasylan®1146), e.g. by using a 1:1 relation, both produced by Evonik Inc. The composition may be further mixed with different amounts of a fine powder of bismuth(III) oxide (>99wt%; $D_{50} \sim 0.3 \mu\text{m}$).

[0062] This good saturation flux density achieved by the material according to the invention makes it possible to downsize inductor components and still maintain good magnetic properties.

10 Compaction and Heat Treatment

[0063] Before compaction the coated iron-based composition may be mixed with a suitable organic lubricant such as a wax, an oligomer or a polymer, a fatty acid based derivate or combinations thereof. Examples of suitable lubricants are EBS, i.e. ethylene bisstearamide, Kenolube® available from Höganäs AB, Sweden, metal stearates such as zinc stearate or fatty acids or other derivatives thereof. The lubricant may be added in an amount of 0.05-1.5% of the total mixture, preferably between 0.1-1.2% by weight.

[0064] Compaction may be performed at a compaction pressure of 400-1200 MPa at ambient or elevated temperature.

[0065] After compaction, the compacted components are subjected to heat treatment at a temperature up to 800°C, preferably between 600-750 °C. Examples of suitable atmospheres at heat treatment are inert atmosphere such as nitrogen or argon or oxidizing atmospheres such as air.

[0066] The powder magnetic core of the present invention is obtained by pressure forming an iron-based magnetic powder covered with a new electrically insulating coating. The core may be characterized by low total losses in the frequency range 2-100 kHz, normally 5-100 kHz, of about less than 12W/kg at a frequency of 20kHz and induction of 0.05T. Further a resistivity, ρ , more than 1000, preferably more than 2000 and most preferably more than 3000 $\mu\Omega\text{m}$, and a saturation magnetic flux density B_s above 1.1, preferably above 1.2 and most preferably above 1.3T. Further, the coercivity shall be below 210A/m, preferably below 200A/m, most preferably below 190A/m and DC- bias not less than 50% at 4000A/m.

30 Examples

[0067] The following examples are intended to illustrate particular embodiments and should not be construed as a limitation of the scope of the invention.

35 Example 1

[0068] Two types of iron powder have been used as core particles; a pure water atomized iron powder having a content of iron above 99.5% by weight and a pure sponge iron having a content of iron above 99.5% by weight. The mean particle size of both types of powder was about 45 μm . The core particles have been mixed with grinded Sendust (typically 85%Fe, 9,5%Si and 5,5%Al) and the powder mix was then treated with a phosphorous containing solution according to WO2008/069749. Briefly, the coating solution was prepared by dissolving 30 ml of 85 % weight of phosphoric acid in 1 000 ml of acetone, and 40 ml - 60 ml of acetone solution was used per 1000 gram of powder. After mixing the phosphoric acid solution with the metal powder, the mixture is allowed to dry.

[0069] The obtained dry phosphorous coated iron - sendust mix powder was further blended with kaolin and sodium silicate according to the following table 1. After drying at 120°C the powder was mixed with 0.6% Kenolube® and compacted at 800MPa into rings with an inner diameter of 45mm, an outer diameter of 55mm and a height of 5mm. The compacted components were thereafter subjected to a heat treatment process at 700°C in a nitrogen atmosphere for 0.5 hours.

[0070] The specific resistivities of the obtained samples were measured by a four point measurement. For maximum permeability, μ_{max} , and coercivity measurements the rings were "wired" with 100 turns for the primary circuit and 100 turns for the secondary circuit enabling measurements of magnetic properties with the aid of a hysteresisgraph, Brockhaus MPG 100. For core loss the rings were "wired" with 30 turns for the primary circuit and 30 turns for the secondary circuit with the aid of Walker Scientific Inc. AMH-401 POD instrument.

[0071] When measuring incremental permeability, the rings were wound with a third winding supplying a DC- bias current of 4 000A/m. DC-bias was expressed as percentage of maximum incremental permeability.

[0072] Unless otherwise stated all tests in the following examples were performed accordingly.

[0073] In order to show the impact of using sponge or atomized iron together with grinded sendust, the impact of a phosphorous coating layer and the impact of the presence of kaolin and sodium silicate in the second coating on the properties of the compacted and heat treated component, samples A-H, were prepared according to table 1 which also

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shows results from testing of the components. In table 1, the invention has also been compared with the use of sponge iron without a first phosphorous coating layer (sample D) according to US4177089.

Table 1

| Sample | Additives on the iron - Sendust mix | | | Component properties | | | | |
|------------------------------------|-------------------------------------|-------------|----------------------|------------------------------------|-----------------|------------------|---------------------------------|--------|
| | Phosphorous coating | Wt-% Kaolin | Wt-% Sodium silicate | Resistivity [$\mu\Omega\cdot m$] | μ_{max} [-] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] | Bs [T] |
| 100%Atomized iron A | | | | | | | | |
| A Comp | Yes | 2% | 0.4% | 20000 | 97 | 222 | 13.5 | 1.98 |
| 100% Sendust B | | | | | | | | |
| B Comp | Yes | 2% | 0.4% | 70000 | 55 | 70 | 5,0 | 0.88 |
| 50%Sponge iron + 50% Sendust C-E | | | | | | | | |
| C Comp | Yes | - | - | 4 | 140 | 249 | 67.8 | 1.42 |
| D Comp | No | 2% | 0.4% | 41 | 87 | 209 | 44.0 | 1.32 |
| E Comp | Yes | 2% | 0.4% | 468 | 76 | 202 | 98 | 1.33 |
| 50%Atomized iron + 50% Sendust F-H | | | | | | | | |
| F Comp | Yes | - | - | 40 | 145 | 180 | 55.2 | 1.52 |
| G Comp | No | 2% | 0.4% | 6013 | 79 | 149 | 9.2 | 1.35 |
| H Inv | Yes | 2% | 0.4% | 77394 | 66 | 138 | 8.2 | 1.35 |

[0074] As can be seen from table 1, the combination of atomized iron, sendust, a primary phosphorous coating layer and a second coating layer consisting of kaolin and sodium silicate considerably improves resistivity and hence lowers core losses. It also gives a good saturation flux density in comparison with 100% sendust.

Example 2

[0075] To illustrate the possibility to dope pure phosphorous and kaolin - sodium silicate coated atomised iron powder with Sendust with only the first phosphorous coating layer and considerably enhance the magnetic properties of the compacted component the following samples were prepared. Table 2 also shows results from testing of the components.

Table 2

| Sample | Additives on Sendust | | | Component properties | | |
|--|----------------------|-------------|----------------------|------------------------------------|------------------|---------------------------------|
| | Phosphorous coating | Wt-% Kaolin | Wt-% Sodium silicate | Resistivity [$\mu\Omega\cdot m$] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] |
| 50%Atomized iron phosphorous and kaolin - sodium silicate coated + 50% Sendust I-L | | | | | | |
| I Comp | No | - | - | 2531 | 169 | 9.5 |
| J Comp | No | 2% | 0.4% | 4587 | 140 | 9.0 |
| K Inv | Yes | - | - | 50354 | 137 | 8.7 |
| L Inv | Yes | 2% | 0.4% | 77394 | 138 | 8.2 |

[0076] As can be seen from table 2, it is beneficial to have a first phosphorous coating layer on the sendust particles.

Example 3

[0077] It is possible to control the magnetic properties of the compacted and heat treated component by varying the content of sendust in the atomized iron powder. The following samples have all been treated in the same way - a first layer of phosphorous coating and a second layer coating consisting of 2%kaolin clay and 0.4%sodium silicate, compacted to 800MPa and heat treated in 700°C for 0.5h in a nitrogen atmosphere. The difference between the samples is that the sendust content in the atomized iron powder has been varied. Table 3 also shows results from testing of the components.

Table 3

| Sample | Wt-% Sendust | Component properties | | | | |
|--|--------------|------------------------------------|-----------------|------------------|---------------------------------|--------|
| | | Resistivity [$\mu\Omega\cdot m$] | μ_{max} [-] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] | Bs [T] |
| Atomized iron + Sendust- different compositions, phosphorous coated and a second coating layer with 2%kaolin and 0.4%sodium silicate M-U | | | | | | |
| M Comp | - | 20000 | 97 | 222 | 13.5 | 1.98 |
| N Inv | 2.5% | 24588 | 91 | 216 | 13.0 | 1.95 |
| O Inv | 5% | 52794 | 87 | 210 | 12.5 | 1.90 |
| P Inv | 10% | 51438 | 85 | 202 | 11.1 | 1.85 |
| Q Inv | 20% | 113513 | 79 | 179 | 10.6 | 1.63 |
| R Inv | 30% | 103656 | 75 | 167 | 9.8 | 1.52 |
| S Inv | 40% | 686475 | 67 | 153 | 9.4 | 1.42 |
| U Inv | 60% | 430569 | 61 | 125 | 7.7 | 1.22 |

[0078] As can be seen from table 3, even a small amount of sendust in the atomized iron powder enhances the magnetic properties of the compacted and heat treated component.

Example 4

[0079] This example shows that the phosphorous- clay- silicate coating concept according to the invention may be applied to different particle sizes of the iron powder - the Sendust powder has a fixed particle size of approximately 45 μm . For sample V) an iron powder having a mean particle size of ~45 μm has been used, for sample W) an iron powder having a mean particle size of ~100 μm has been used and for sample X) an iron powder having a mean particle size of ~210 μm has been used. The iron - Sendust powder mix was coated with a first phosphorous containing layer. Thereafter some samples were further treated with 1% kaolin and 0.4% sodium silicate as earlier described. Heat treatment was performed for 0.5h at 700°C in nitrogen.

[0080] Results from testing of samples V-X) according to table 4

Table 4

| Sample | Powder mix properties | | | | Component properties | | |
|--|--------------------------------|-------------|----------------------|--------------|------------------------------------|------------------|---------------------------------|
| | Mean particle size [μm] | Wt-% Kaolin | Wt-% Sodium silicate | Wt-% Sendust | Resistivity [$\mu\Omega\cdot m$] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] |
| All samples have a first phosphorous coating layer V-X | | | | | | | |
| V Inv | 45 | 1% | 04% | 40% | 72850 | 134 | 75 |
| Sample V Comp | 45 | 1% | 0.4% | - | 15000 | 226 | 15.0 |
| W Inv | 100 | 1% | 0.4% | 40% | 88187 | 105 | 9.9 |

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(continued)

| Sample | Powder mix properties | | | | Component properties | | |
|---------------|--------------------------------------|-------------|----------------------|--------------|--|------------------|---------------------------------|
| | Mean particle size [μm] | Wt-% Kaolin | Wt-% Sodium silicate | Wt-% Sendust | Resistivity [$\mu\Omega\cdot\text{m}$] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] |
| Sample W Comp | 100 | 1% | 0.4% | - | 19000 | 177 | 25.2 |
| X Inv | 210 | 1% | 0.4% | 40% | 114479 | 83 | 11.1 |
| Sample X Comp | 210 | 1% | 0.4% | - | 35000 | 140 | 30.1 |

[0081] Table 4 shows that regardless of the particle size of the iron powder clear improvements of resistivity and core losses are obtained for components according to the present invention.

Example 5

[0082] Example 5 illustrates that it is possible to use different types of water glass and different types of clays containing defined phyllosilicates. The 60% atomized iron - 40% sendust powder mixes were coated as described above with the exception that various silicates (Na, K and Li) and various clays (kaolin and talc) containing phyllosilicates having electroneutral layers were used. In comparative examples clays containing phyllosilicates having electrical charged layer, Veegum® and a mica, were used. Veegum® is a trade name of clay from the smectite group. The mica used was muscovite. The second layer in all the tests contained 1% of clay and 0.4wt-% of water glass. Heat treatment was performed for 0.5h at 700°C in nitrogen.

[0083] The following table 5 shows results from testing of the components.

Table 5

| Sample | Additives on the iron - Sendust mix | | | Component properties | | | |
|--|-------------------------------------|------------------|--------------------|--|------------------------|------------------|--------------------------------|
| | Type of clay | Type of silicate | Mol ratio silicate | Resistivity [$\mu\Omega\cdot\text{m}$] | μ_{max} [-] | Coercivity [air] | Coreloss at 0.05T 20kHz [W/kg] |
| 60%Atomized iron + 40% Sendust- phosphorous coated with a second coating layer consisting of 1%clay and 0.4%silicate Y-e | | | | | | | |
| Y Inv | Kaolin | Na | 2.5 | 72850 | 80 | 134 | 7.5 |
| Z Inv | Talc | Na | 2.5 | 72321 | 94 | 131 | 7.4 |
| a Comp | Veegum® | Na | 2.5 | 97 | 91 | 135 | 19.9 |
| b Comp | Mica | Na | 2.5 | 389 | 106 | 138 | 15.4 |
| c Inv | Kaolin | Na | 3.37 | 72569 | 84 | 136 | 7.3 |
| d Inv | Kaolin | K | 2.5 | 84992 | 86 | 140 | 8.4 |
| e Inv | Kaolin | Li | 2.5 | 77403 | 85 | 147 | 8.2 |

[0084] As evident from table 5, various types of water glass and clays containing defined phyllosilicates can be used provided the phyllosilicate is of the type having electroneutral layers.

Example 6

[0085] Example 6 illustrates that by varying the amounts of clay and alkaline silicate in the second layer the properties of the compacted and heat treated component can be controlled and optimized. The samples were prepared and tested as described earlier. Heat treatment was performed for 0.5h at 700°C in nitrogen.

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[0086] The following table 6 shows results from testing

Table 6

| Sample | Additives on the iron - Sendust mix | | Component properties | | |
|--|-------------------------------------|----------------------|------------------------------------|------------------|---------------------------------|
| | Wt-% Kaolin | Wt-% Sodium silicate | Resistivity [$\mu\Omega\cdot m$] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] |
| 60%Atomized iron + 40% Sendust, phosphorous coated f-n | | | | | |
| f Inv | - | 0.4% | 100 | 151 | 15.8 |
| g Inv | 0.5% | 0.4% | 2956 | 155 | 10.2 |
| h Inv | 1% | 0.4% | 172850 | 154 | 9.6 |
| i Inv | 2% | 0.4% | 686475 | 153 | 9.4 |
| J Inv | 3% | 0.4% | 732463 | 157 | 10.0 |
| k Inv | 5% | 0.4% | 179478 | 164 | 11.6 |
| l Inv | 2% | 0.2% | 136795 | 157 | 10.1 |
| m Inv | 2% | 0.6% | 88309 | 156 | 10.1 |
| n Inv | 2% | 08% | 3359 | 164 | 10.3 |

[0087] As can be seen from table 6, resistivity will decrease if the content of sodium silicate in the second layer exceeds 0.7% by weight. Resistivity will also decrease as the content of sodium silicate is decreased thus the content of silicate shall be between 0.2-0.7% by weight, preferably between 0.3-0.6 % by weight of the total 60%atomized iron - 40% Sendust powder mix. Further increased clay content in the second layer up to about 4% will increase resistivity but decrease core loss due to increased Coercivity. Thus, the upper limit of clay in the second layer is 5 %, preferably 4%, by weight of the iron- based composite powder. The lower limit for content of clay is 1 %, preferably 3% as a too low content of clay will have a detrimental influence of resistivity and core loss.

Example 7

[0088] The following example 7 illustrates that powder produced according to the invention can be compacted to different compaction pressures and at different compaction die temperatures. The samples below have been treated as described above, 60%atomized iron and 40% Sendust has been phosphorous and clay - sodium silicate coated, the content of kaolin in the second layer was 2% and the content of sodium silicate was 0.4% by weight of the composite iron - Sendust powder.

[0089] The samples o-v) were compacted to between 400 - 1200MPa either at 20°C or 60°C compaction die temperature and heat treated for 0.5h at 700°C in nitrogen. Results from testing according to table 7;

Table 7

| Sample | Compaction properties | | Component properties | | | | | Bs [T] |
|--|---------------------------|---------------------------------|----------------------|------------------------------------|-----------------|------------------|---------------------------------|--------|
| | Compaction pressure [MPa] | Compaction die temperature [°C] | Density [g/cc] | Resistivity [$\mu\Omega\cdot m$] | μ_{max} [-] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] | |
| 60%Atomized iron + 40% Sendust - phosphorous coated with a second coating layer consisting of 2%kaolin and 0.4%sodium silicate o-v | | | | | | | | |
| o Inv | 400 | 20 | 5.86 | 25441 | 56 | 167 | 10.0 | 1 30 |
| p Inv | 600 | 20 | 6.15 | 52357 | 65 | 166 | 9.3 | 1 36 |
| q Inv | 800 | 20 | 6.27 | 686475 | 67 | 153 | 9.4 | 1 42 |
| r Inv | 1000 | 20 | 6.41 | 773125 | 79 | 166 | 8.6 | 1 41 |
| s Inv | 1000 | 60 | 6.42 | 720625 | 86 | 156 | 83 | 1.42 |

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(continued)

| Sample | Compaction properties | | Component properties | | | | | |
|--------|---------------------------|---------------------------------|----------------------|------------------------------------|-----------------|------------------|---------------------------------|--------|
| | Compaction pressure [MPa] | Compaction die temperature [°C] | Density [g/cc] | Resistivity [$\mu\Omega\cdot m$] | μ_{max} [-] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] | Bs [T] |
| t Inv | 1100 | 20 | 643 | 796750 | 83 | 165 | 8.4 | 1.43 |
| u Inv | 1100 | 60 | 6.45 | 101250 | 86 | 166 | 8.3 | 1.44 |
| v Inv | 1200 | 60 | 6.50 | 96875 | 90 | 162 | 81 | 1.44 |

[0090] Table 7 shows that high resistivity and low core losses are obtained for components, according to the invention, compacted to different compaction pressures and compacted at different compaction die temperatures. A rise of the density and a lowering of the losses can be observed when raising the compaction pressure from 400 to 800MPa, further increasing the compaction pressure however gives just little effect. The compaction die temperature only increases the resistivity slightly and does not give any further improvements of the magnetic properties.

Example 8

[0091] The following example 8 illustrate that components produced from powder according to the invention can be heat treated in different atmospheres and different temperatures. The samples below have been treated as described above, 60%atomized iron and 40% Sendust has been phosphorous- and clay - sodium silicate coated, the content of kaolin in the second layer was 2% and the content of sodium silicate was 0.4% by weight of the composite iron - Sendust powder.

[0092] The samples w-Dd) were heat treated at between 550 - 750°C in nitrogen and air respectively. Results from testing according to table 8;

Table 8

| Sample | Heat treatment parameters | | Component properties | | | |
|---|---------------------------------|---------------------------|------------------------------------|-----------------|------------------|---------------------------------|
| | Heat treatment temperature [°C] | Heat treatment atmosphere | Resistivity [$\mu\Omega\cdot m$] | μ_{max} [-] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] |
| 60%Atomized iron + 40% Sendust - phosphorous coated with a second coating layer consisting of 2%kaolin and 0.4%sodium silicate w-Dd | | | | | | |
| w Comp | 550°C | Nitrogen | 190574 | 54 | 283 | 22.8 |
| x Comp | 600°C | Nitrogen | 184382 | 61 | 228 | 16.6 |
| y Inv | 650°C | Nitrogen | 178613 | 69 | 183 | 12.1 |
| z Inv | 700°C | Nitrogen | 686475 | 67 | 153 | 9.4 |
| Aa Inv | 750°C | Nitrogen | 566356 | 70 | 150 | 8.9 |
| Bb Inv | 700°C | 90%Nitrogen + + 10%Air | 561917 | 69 | 165 | 9.2 |
| Cc Inv | 700°C | 50%Nitrogen + 50%Air | 429138 | 66 | 250 | 10.5 |
| Dd Comp | 700°C | Air | 17400 | 64 | 303 | 12.1 |

[0093] Table 8 shows that high resistivity and low core losses are obtained for components according to the invention heat treated at between 650°C - 750°C in nitrogen atmosphere or in a mixed atmosphere with nitrogen and air.

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Example 9

[0094] The following example 9 illustrates that it is possible to boost the magnetic properties of components produced from powder according to the invention by adding gas atomized FeSi to the mix. The iron - Sendust powder mixes have a first phosphorous coating layer and a second layer consisting of 2%kaolin and 0.4%sodium silicate. The powder mixes have been compacted to 800MPa and heat treated at 700°C, for 30minutes in a nitrogen atmosphere.

Table 9

| Sample | Mixture composition | | Component properties | | | |
|---|---------------------|------------------------|------------------------------------|-----------------|------------------|---------------------------------|
| | Wt-% Sendust | Wt-% Gas atomized FeSi | Resistivity [$\mu\Omega\cdot m$] | μ_{max} [-] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] |
| Atomized iron powder - the powder mixtures have a phosphorous coating and a second coating layer consisting of 2%kaolin and 0.4%sodium silicate Ee-Gg | | | | | | |
| Ee Inv | 30% | - | 103656 | 75 | 167 | 9.8 |
| Ff Inv | 40% | - | 686475 | 67 | 153 | 9.4 |
| Gg Inv | 30% | 10% | 704380 | 55 | 149 | 8.7 |

[0095] As can be seen in table 9 an adding of 10%FeSi in the phosphorus and kaolin - sodium silicate coated iron - Sendust mix enhances the resistivity and lowers the Coercivity and the core losses.

Example 10

[0096] A pure water atomized iron powder having a content of iron above 99.5% by weight has been used as core particles. The mean particle size of the powder was about 45 μm . The core particles have been mixed with Sendust (typically 85%Fe, 9%Si and 6%Al) and the powder mix was treated with a phosphorous containing solution according to WO2008/069749. The obtained dry phosphorous coated iron powder-sendust mix was further treated with a second (metal organic) coating layer as described in WO2009/116938, namely mixing the powder by stirring with different amounts of first a basic aminoalkyl-alkoxy silane (Dynasylan®Ameo) and thereafter with an oligomer of an aminoalkyl/alkyl-alkoxy silane (Dynasylan®1146), using a 1:1 relation, both produced by Evonik Inc. The composition was further mixed with different amounts of a fine powder of bismuth(III) oxide (>99wt%; $D_{50} \sim 0.3 \mu m$).

[0097] After coating the powder was mixed with 0.4% amide wax and compacted to 800 MPa into rings with an inner diameter of 45mm, an outer diameter of 55mm and a height of 5mm. The compacted components were thereafter subjected to a heat treatment process at 700°C in a nitrogen atmosphere for 0.5 hours.

[0098] Unless otherwise stated all tests in the following examples were performed accordingly.

[0099] Samples Hh-li) were prepared according to table 10 which also shows results from testing of the components.

Table 10

| Sample | Additives on the iron -Sendust mix | | Heat treatment temperature | Component properties | | | | |
|-----------------------------------|------------------------------------|---------------------|----------------------------|------------------------------------|-----------------|------------------|---------------------------------|-------------|
| | Phosphorous coating | Metal organic layer | | Resistivity [$\mu\Omega\cdot m$] | μ_{max} [-] | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] | DC Bias [%] |
| 100%Atomized Iron Hh | | | | | | | | |
| Hh Comp | Yes | Yes | 650°C | 19923 | 226 | 195 | 10,0 | 40 |
| 60%Atomized Iron + 40% Sendust li | | | | | | | | |
| li Inv | Yes | Yes | 700°C | 125000 | 113 | 151 | 6.7 | 60 |

[0100] As can be seen from table 10 the combination of atomized iron, Sendust, a primary phosphorous coating layer

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and a second (metal organic) coating layer considerably improves resistivity, DC-bias and lowers core losses and Coercivity compared to using 100%atomized iron powder.

Example 11

[0101] It is possible to control the magnetic properties of the compacted and heat treated component by varying the content of sendust in the atomized iron powder. The following samples have all been treated in the same way - a first layer of phosphorous coating and a second (metal organic) coating layer. The difference between the samples is that the sendust content in the atomized iron powder has been varied. The samples have all been compacted to 800MPa and heat treated for 0.5h at 700°C in a nitrogen atmosphere. Table 11 also shows results from testing of the components.

Table 11

| Sample | Composition | | Component properties | | | | |
|---|--------------|-----------|----------------------|------------------|---------------------------------|-------------|--------|
| | Wt-% Sendust | Wt-% Iron | $\mu_{max}[-]$ | Coercivity [A/m] | Core loss at 0.05T 20kHz [W/kg] | DC-Bias [%] | Bs [T] |
| Atomized iron + Sendust - different compositions - phosphorous coated with a second layer metal organic coating | | | | | | | |
| Jj Comp | - | 100% | 226 | 195 | 10 | 40 | 1.99 |
| Kk Inv | 20% | 80% | 135 | 176 | 6.9 | 51 | 1.72 |
| Ll Inv | 30% | 70% | 123 | 164 | 6.8 | 55 | 1.61 |
| Mm Inv | 40% | 60% | 113 | 151 | 6.7 | 60 | 1.48 |
| Nn Inv | 50% | 50% | 104 | 139 | 6.6 | 63 | 1.35 |

[0102] As for the clay/sodium silicate coated atomized iron- and sendust-powder-mix an increased share of sendust considerably improves resistivity and DC-bias and hence lowers core losses and Coercivity.

Claims

1. A composite iron-based powder composition comprising core particles wherein the core particles are a mixture of
 - (a) iron alloy particles consisting essentially of 7% to 13% by weight silicon, 4% to 7% by weight aluminium, the balance being iron, and
 - (b) atomized iron particles, and wherein said core particles are coated with a first phosphorous containing layer.
2. Composite iron-based powder according to claim 1, wherein the atomized iron particles have a second layer comprising;
 - (a) an alkaline silicate combined with a clay mineral containing a phyllosilicate, the combined silicon-oxygen tetrahedral layer and hydroxide octahedral layers thereof being electrical neutral, or;
 - (b) a metal organic layer.
3. Composite iron-based powder according to claim 2, wherein the iron alloy particles have a second layer comprising;
 - a) an alkaline silicate combined with a clay mineral containing a phyllosilicate, the combined silicon-oxygen tetrahedral layer and hydroxide octahedral layers thereof being electrical neutral, or;
 - b) a metal organic layer.
4. Composite iron-based powder according to claim 3, wherein the iron alloy particles and the atomized iron particles have the same second layer.
5. A composite iron-based powder composition according to any one of claims 2-4, wherein the second layer comprises kaolin and sodium silicate.

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6. A composite iron-based powder composition according to any one of claims 1-5, further comprising atomized FeSi.
7. A composite iron-based powder composition according to any one of claims 1 to 6, wherein the phosphorous containing layer has a thickness between 20 and 300 nm.
- 5
8. A composite iron-based powder according to any one of claims 1-7, wherein the phosphorous coating is provided by contacting the core particles with a phosphorous compound in a solvent and afterwards removing the solvent by drying.
- 10
9. A composite iron-based powder according to any one of claims 1-8, wherein the phosphorous compound is phosphoric acid or ammoniumphosphate.
10. The composite iron-based powder according to any one of claims 2-9, wherein the content of alkaline silicate is between 0.1-0.9% by weight, preferably between 0.2-0.8% by weight of the composite iron-based powder.
- 15
11. The composite iron based powder according to any one of claims 2-10, wherein the content of clay is between 0.2-5% by weight, preferably between 0.5-4 % by weight of the composite iron- based powder.
12. A method for producing a compacted and heat treated component comprising the steps of:
- 20
- a) providing a composite iron-based powder according to any of claims 1-11,
 - b) compacting the composite iron-based powder, optionally mixed with a lubricant, in a uniaxial press movement in a die at a compaction pressure between 400 and 1200 MPa
 - c) ejecting the compacted component from the die.
 - 25 d) heat treating the ejected component in a non-reducing atmosphere at a temperature up to 800°C.
13. A component produced according to the method described in claim 12.
14. An inductor core produced according to the method of claim 13.
- 30
15. An inductor core according to claim 14, having a resistivity, ρ , above 1000, preferably above 2000 and most preferably above 3000 $\mu\Omega\text{m}$; a saturation magnetic flux density B_s above 1.1, preferably above 1.2 and most preferably above 1.3 (T); ; core loss less than 12W/kg at a frequency of 20kHz; induction of 0.05T; coercivity below 210A/m, preferably below 200A/m, most preferably below 190A/m; and DC-bias not less than 50% at 4000A/m.
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EUROPEAN SEARCH REPORT

 Application Number
 EP 11 16 1536

| DOCUMENTS CONSIDERED TO BE RELEVANT | | | |
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| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | CLASSIFICATION OF THE APPLICATION (IPC) |
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| A | US 2007/144614 A1 (LU ZHICHAO [CN] ET AL) 28 June 2007 (2007-06-28) * paragraphs [0083] - [0085]; claims 1-33 * | 1-15 | |
| The present search report has been drawn up for all claims | | | |
| Place of search The Hague | | Date of completion of the search 21 September 2011 | Examiner Primus, Jean-Louis |
| CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document | | T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document | |

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EPC FORM 1503 03 02 (P04C01)



CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing claims for which payment was due.

- Only part of the claims have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due and for those claims for which claims fees have been paid, namely claim(s):
- No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due.

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

see sheet B

- All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.
- As all searchable claims could be searched without effort justifying an additional fee, the Search Division did not invite payment of any additional fee.
- Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid, namely claims:
- None of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:
- The present supplementary European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims (Rule 164 (1) EPC).



**LACK OF UNITY OF INVENTION
SHEET B**

Application Number
EP 11 16 1536

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

1. claims: 1, 4-15(completely); 2, 3(partially)

composite phosphate insulated iron-based powder composition, a corresponding component and its production method and a corresponding inductor core wherein said powder is further insulated with an alkaline silicate combined with a clay mineral

2. claims: 1, 4-15(completely); 2, 3(partially)

composite phosphate insulated iron-based powder composition, a corresponding component and its production method and a corresponding inductor core wherein said powder is further insulated with a metal organic layer

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 11 16 1536

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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21-09-2011

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