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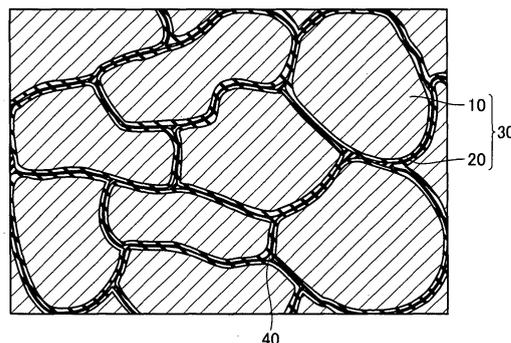
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(54) **SOFT MAGNETIC MATERIAL AND METHOD FOR PRODUCING SAME**

(57) Disclosed are a soft magnetic material having an optimum electrical resistivity and a method for producing such a soft magnetic material. The soft magnetic material comprises a plurality of composite magnetic particles (30). Each of the composite magnetic particles (30) comprises a metal magnetic particle (10) and an insulating coating film (20) which covers the surface of the metal magnetic particle (10) and contains at least one substance selected from the group consisting of zirconium

oxide, aluminum oxide and silicon oxide. The soft magnetic material has an electrical resistivity of not less than 3,000  $\mu\Omega\text{cm}$  and not more than 50,000  $\mu\Omega\text{cm}$ , and a magnetic permeability  $\mu$  of not less than 2,000 and not more than 4,000. A method for producing such a soft magnetic material comprises a step for pressure forming a compaction by compressing a plurality of the composite magnetic particles (30) and a step for subjecting the compaction to a first heat treatment at not less than 400°C and not more than 900°C.

FIG.1



## Description

### Technical Field

[0001] The present invention relates to a soft magnetic material and a method for producing the same, and more specifically, it relates to a soft magnetic material comprising composite magnetic particles having metal magnetic particles and insulating coating films and a method for producing the same.

### Background Art

[0002] Electrical/electronic components have recently been densified and downsized, and capability of performing more precise control with saved power is demanded in relation to motor cores and transformer cores. Therefore, development of a soft magnetic material, used for these electrical/electronic components, having excellent magnetic characteristics in intermediate and high frequency domains is in progress. In order to exhibit excellent magnetic characteristics in the intermediate and high frequency domains, the soft magnetic material must have high saturation magnetic flux density, high magnetic permeability and high electric resistivity.

[0003] Japanese Patent Laying-Open No. 6-267723 (patent literature 1), for example, discloses such a soft magnetic material.

[0004] Patent Literature 1: Japanese Patent Laying-Open No. 6-267723

### Disclosure of the Invention

#### Problems to be Solved by the Invention

[0005] In the soft magnetic material disclosed in the aforementioned literature, however, there has been such a problem that electric resistivity is excessively high and magnetic flux density is small.

[0006] Accordingly, the present invention has been proposed in order to solve the aforementioned problem, and an object of the present invention is to provide a soft magnetic material having optimized electric resistivity and a method for producing the same.

#### Means for Solving the Problems

[0007] The soft magnetic material according to the present invention comprises a plurality of composite magnetic particles. Each of the plurality of composite magnetic particles has a metal magnetic particle and an insulating coating film, containing at least one substance selected from a group consisting of aluminum oxide, zirconium oxide and silicon oxide, surrounding the surface of the metal magnetic particle. Electric resistivity  $\rho$  of the soft magnetic material is at least 3000  $\mu\Omega\text{cm}$  and not more than 50000  $\mu\Omega\text{cm}$ .

[0008] More preferably, magnetic permeability  $\mu$  of the

soft magnetic material is at least 2000 and not more than 4000. The method for producing a soft magnetic material according to the present invention is a method for producing the aforementioned soft magnetic material and comprises the steps of preparing a compaction by pressing a plurality of composite magnetic particles having metal magnetic particles and insulating coating films, containing at least one substance selected from a group consisting of aluminum oxide, zirconium oxide and silicon oxide, surrounding the surfaces of the metal magnetic particles and performing first heat treatment on the compaction at a temperature of at least 400°C and not more than 900°C.

[0009] Preferably, the method for producing a soft magnetic material further comprises the step of pressing the compaction after the first heat treatment and thereafter performing second heat treatment on the compaction under the atmospheric pressure at a temperature of at least 400°C and not more than 900°C.

### Effects of the Invention

[0010] According to the present invention, a soft magnetic material having desired magnetic characteristics and a method for producing the same can be provided.

### Brief Description of the Drawings

[0011]

Fig. 1 is a typical diagram showing a section of a soft magnetic material according to an embodiment of the present invention.

35 Description of the Reference Signs

[0012] 10 metal magnetic particle, 20 insulating coating film, 30 composite magnetic particle.

40 **Best Modes for Carrying Out the Invention**

[0013] A soft magnetic material according to the present invention has a plurality of composite magnetic particles, and each of the composite magnetic particles has a metal magnetic particle and an insulating coating film surrounding the surface of the metal magnetic particle.

[0014] The metal magnetic particles are generally made of iron (Fe). However, the metal magnetic particles are not restricted to iron, but may alternatively be formed by other magnetic particles. For example, the metal magnetic particles may be made of an iron (Fe)-silicon (Si) alloy, an iron (Fe)-nitrogen (N) alloy, an iron (Fe)-nickel (Ni) alloy, an iron (Fe)-carbon (C) alloy, an iron (Fe)-boron (B) alloy, an iron (Fe)-cobalt (Co) alloy, an iron (Fe)-phosphorus (P) alloy, an iron (Fe)-nickel (Ni)-cobalt (Co) alloy or an iron (Fe)-aluminum (Al)-silicon (Si) alloy. The metal magnetic particles may be of a simple substance of metal

or an alloy.

**[0015]** The average particle diameter of the metal magnetic particles is preferably at least 5  $\mu\text{m}$  and not more than 200  $\mu\text{m}$ . If the average particle diameter of the metal magnetic particles is less than 5  $\mu\text{m}$ , the metal is so easily oxidized that the magnetic characteristics of the soft magnetic material may be reduced. If the average particle diameter of the metal magnetic particles exceeds 20  $\mu\text{m}$ , compressibility of mixed powder is reduced in a subsequent pressure-forming step. Thus, the density of a compaction obtained through the pressure-forming step may be so reduced that it is difficult to handle the compaction.

**[0016]** It is to be noted that the average particle size described herein refers to a particle size obtained when the sum of masses of particles added in ascending order of particle size in a histogram of particle sizes measured by sieving reaches 50 % of the total mass, that is, 50 % particle size D.

**[0017]** The insulating coating films can be made of an oxide insulator containing aluminum and/or zirconium and/or silicon. The electric resistivity  $\rho$  of the soft magnetic material can be increased by covering the surfaces of the metal magnetic particles with the insulating coating films. Thus, iron loss of the soft magnetic material resulting from eddy current can be reduced by inhibiting the eddy current from flowing between the metal magnetic particles.

**[0018]** According to the present invention, the electric resistivity  $\rho$  of the soft magnetic material is at least 3000  $\mu\Omega\text{cm}$  and not more than 50000  $\mu\Omega\text{cm}$ . If the electric resistivity  $\rho$  is less than 3000  $\mu\Omega\text{cm}$ , the electric resistivity is reduced to reduce the effect of suppressing the eddy current.

**[0019]** If the electric resistivity  $\rho$  exceeds 50000  $\mu\Omega\text{cm}$  contrarily thereto, the electric resistivity is unpreferably excessively increased. More specifically, increase of the electric resistivity  $\rho$  means increase of the quantity of the insulating coating films. If the quantity of the insulating coating films is excessively increased, magnetic characteristics such as magnetic permeability and magnetic flux density are deteriorated.

**[0020]** In order to improve the aforementioned effect, the electric resistivity  $\rho$  of the soft magnetic material is preferably at least 6000  $\mu\Omega\text{cm}$  and not more than 15000  $\mu\Omega\text{cm}$ , more preferably at least 8000  $\mu\Omega\text{cm}$  and not more than 10000  $\mu\Omega\text{cm}$ .

**[0021]** The thickness of the insulating coating films is preferably at least 0.005  $\mu\text{m}$  and not more than 20  $\mu\text{m}$ . Energy loss resulting from eddy current can be effectively suppressed by setting the thickness of the insulating coating films to at least 0.005  $\mu\text{m}$ . When the thickness of the insulating coating films is set to not more than 20  $\mu\text{m}$ , the volume ratio of the insulating coating films occupying the soft magnetic material is not excessively increased. Thus, a soft material having prescribed saturation magnetic flux density can be formed.

**[0022]** More preferably, magnetic permeability  $\mu$  of the soft magnetic material is at least 2000 and not more than

4000. Further preferably, the magnetic permeability  $\mu$  of the soft magnetic material is at least 2500 and not more than 3500.

**[0023]** A method for producing the aforementioned soft magnetic material is now described. First, a plurality of composite magnetic particles are prepared. These composite magnetic particles are introduced into a powder compaction, and mixed powder is pressure-formed under a condition of pressure of at least 390 MPa and not more than 1500 MPa, for example. Thus, the mixed powder is so compressed that a compaction can be obtained. The pressure forming is preferably performed under an inert gas atmosphere or a decompressed atmosphere. In this case, the mixed powder can be prevented from oxidation by oxygen in the atmosphere. In the step of preparing the compaction, well-known warm pressing or die wall lubrication is so employed as to densify the compaction, improve the space factor and improve the magnetic characteristics. The powder temperature in the warm pressing is preferably 100°C to 180°C.

**[0024]** In order to reinforce bonding between the composite magnetic particles, organic matter may intervene between the composite magnetic particles. In this case, the composite magnetic particles and the organic matter must be previously mixed with each other. The mixing method is not restricted but any of mechanical alloying, vibration ball milling, satellite ball milling, mechanofusion, coprecipitation, chemical vapor deposition (CVD), physical vapor deposition (PVD), plating, sputtering, vapor deposition and a sol-gel process can be used.

**[0025]** Thermoplastic resin such as thermoplastic polyimide, thermoplastic polyamide, thermoplastic polyamidimide, polyphenylene sulfide, polyamidimide, poly(ethersulfone), polyether imide or poly(etheretherketone) can be employed for the organic matter. This organic matter is so provided that the organic matter functions as a lubricant between the plurality of composite magnetic particles. Thus, breakage of the insulating coating films can be suppressed in the pressure-forming step.

**[0026]** Then, the compaction obtained by the pressure forming is heat-treated at a temperature of at least 400°C and not more than 900°C. Large numbers of strains and dislocations are caused in the compaction obtained through the pressure-forming step, and the strains and the dislocations result in reduction of the magnetic permeability and increase of the coercive force. The heat treatment is performed on the compaction, in order to eliminate these strains and dislocations. Such heat treatment is required also when the organic matter intervenes between the composite magnetic particles.

**[0027]** In order to improve the density of the soft magnetic material and eliminate the dislocations and the strains from the soft magnetic material, the soft magnetic material is compressed again to be improved in density, and thereafter heat-treated under the atmospheric pressure at a temperature of 400°C and not more than 900°C.

**[0028]** Thus, the inventive soft magnetic material can be produced.

**[0029]** In general, hysteresis loss is reduced if the coercive force of the soft magnetic material is small, the coercive force is also increased if the hysteresis loss is large, and the magnetic permeability is increased if the coercive force is small. Improvement of the magnetic permeability leads to reduction of the hysteresis loss. According to the present invention, the material is so constituted as to increase the magnetic permeability, leading to reduction of the hysteresis loss.

**[0030]** In order to reduce eddy current loss, it is important to keep insulation between the composite magnetic particles. Increase of specific resistance of the soft magnetic material as a bulk body leads to reduction of the eddy current loss. In particular, the eddy current loss includes eddy current loss in the respective particles and eddy current loss caused between the particles. The eddy current loss between the particles must be reduced, and it is possible to reduce the eddy current loss according to the present invention since the specific resistance of the soft magnetic material is increased in the range not damaging the magnetic characteristics.

**[0031]** The compositions of aluminum oxide, zirconium oxide and silicon oxide constituting the insulating coating films in the present invention are not particularly restricted. More specifically, the composition of aluminum oxide is not restricted to  $Al_2O_3$ , and the atomic ratio between aluminum and oxygen may be properly changed. Also as to the composition ratio of zirconium oxide, the ratio between zirconium and oxygen may be properly changed. Further, the ratio between silicon and oxygen may be properly changed also as to the composition ratio of silicon oxide.

**[0032]** Fig. 1 is a schematic diagram showing a section of a soft magnetic material according to an embodiment of the present invention. Referring to Fig. 1, the soft magnetic material comprises a plurality of composite magnetic particles 30. Each of the plurality of composite magnetic particles 30 has a metal magnetic particle 10 and an insulating coating film 20, containing at least one substance selected from a group consisting of aluminum oxide, zirconium oxide and silicon oxide, surrounding the surface of the metal magnetic particle 10. The electric resistivity  $\rho$  of the soft magnetic material is at least 3000  $\mu\Omega\text{cm}$  and not more than 50000  $\mu\Omega\text{cm}$ . Organic matter 40 intervenes between the composite magnetic particles 30.

(Example 1)

**[0033]** According to Example 1, a soft magnetic material according to the present invention was produced. First, iron particles having an average particle diameter of 70  $\mu\text{m}$  were prepared as metal magnetic particles. These iron particles were coated with  $Al_2O_3$  films serving as insulating coating films by a wet method. At this time, the thickness of the insulating coating films was set to about 100 nm. Composite magnetic particles were formed by surrounding the surfaces of the iron particles

with the  $Al_2O_3$  films through this coating.

**[0034]** Mixed powder was prepared by mixing the composite magnetic particles and particles of polyphenylene sulfide resin having an average particle diameter of not more than 100  $\mu\text{m}$  with each other. The mixed powder was introduced into a metal mold and subjected to pressure molding. At this time, the pressure molding was performed in a nitrogen gas atmosphere, the metal mold was set to the normal temperature, and the pressure was set to 882 MPa. Thus, a sample of a compaction was obtained. Then, the compaction was heat-treated. The heat treatment was performed in a nitrogen gas atmosphere at a temperature of 800°C for 3 hours. Electric resistivity, density and magnetic permeability  $\mu$  of the sample thereafter measured were 5670  $\mu\Omega\text{cm}$ , 7.5  $\text{g}/\text{cm}^3$  and 2050 respectively.

(Comparative Example)

**[0035]** In comparative example 1, Somalloy 500 (trade name) was prepared as composite magnetic particles. Somalloy 500 is composite magnetic particles prepared by molding phosphate coating films on the surfaces of iron particles. Mixed powder was prepared by mixing particles of polyphenylene sulfide into the composite magnetic particles. The mixed powder was introduced into a compaction and subjected to pressure forming. At this time, the pressure forming was performed in a nitrogen gas atmosphere, the metal mold was set to the normal temperature, and the pressure was set to 882 MPa. Thus, a compaction was obtained.

**[0036]** Then, the compaction was heat-treated. The heat treatment was performed in a nitrogen gas atmosphere at a temperature of 300°C for 0.5 hours. Thereafter electric resistivity and magnetic permeability of the compaction were measured. The electric resistivity was 350  $\mu\Omega\text{cm}$ , and the magnetic permeability  $\mu$  was 600.

**[0037]** From the aforementioned results, it has been confirmed that the inventive soft magnetic material can satisfy the magnetic characteristics required to the soft magnetic material.

**[0038]** The embodiment and Example disclosed this time must be considered as illustrative in all points and not restrictive. The scope of the present invention is shown not by the above description but by the scope of claim for patent, and it is intended that all modifications within the meaning and range equivalent to the scope of claim for patent are included.

## Claims

1. A soft magnetic material comprising a plurality of composite magnetic particles (30), wherein each of said plurality of composite magnetic particles (30) has a metal magnetic particle (10) and an insulating coating film (20), containing at least one substance selected from a group consisting of aluminum

oxide, zirconium oxide and silicon oxide, surrounding the surface of said metal magnetic particle (10), and  
 electric resistivity  $\rho$  is at least 3000  $\mu\Omega\text{cm}$  and not more than 50000  $\mu\Omega\text{cm}$ .

2. The soft magnetic material according to claim 1, wherein magnetic permeability  $\mu$  is at least 2000 and not more than 4000.

3. A method for producing the soft magnetic material according to claim 1 or 2, comprising the steps of:

preparing a compaction by pressing a plurality of composite magnetic particles (30) having metal magnetic particles (10) and insulating coating films (20), containing at least one substance selected from a group consisting of aluminum oxide, zirconium oxide and silicon oxide, surrounding the surfaces of said metal magnetic particles (10); and  
 performing first heat treatment on said compaction under the atmospheric pressure at a temperature of at least 400°C and not more than 900°C.

4. The method for producing a soft magnetic material according to claim 3, further comprising the step of pressing said compaction after performing said first heat treatment and thereafter performing second heat treatment under the atmospheric pressure at a temperature of at least 400°C and not more than 900°C.

of composite magnetic particles (30) having metal magnetic particles (10) and insulating coating films (20), containing at least one substance selected from a group consisting of aluminum oxide, zirconium oxide and silicon oxide, surrounding the surfaces of said metal magnetic particles (10); and  
 performing first heat treatment on said compaction under the atmospheric pressure at a temperature of at least 400°C and not more than 900°C.

4. The method for producing a soft magnetic material according to claim 3, further comprising the step of pressing said compaction after performing said first heat treatment and thereafter performing second heat treatment under the atmospheric pressure at a temperature of at least 400°C and not more than 900°C.

#### Amended claims under Art. 19.1 PCT

1. (Amended) A soft magnetic material comprising a plurality of composite magnetic particles (30), wherein  
 each of said plurality of composite magnetic particles (30) has a metal magnetic particle (10) and an insulating coating film (20), containing at least one substance selected from a group consisting of aluminum oxide, zirconium oxide and silicon oxide, surrounding the surface of said metal magnetic particle (10), and  
 electric resistivity  $\rho$  is at least 3000  $\mu\Omega\text{cm}$  and not more than 50000  $\mu\Omega\text{cm}$ , and magnetic permeability  $\mu$  is at least 2000 and not more than 4000.

2. (cancelled)

3. (Amended) A method for producing the soft magnetic material according to claim 1, comprising the steps of:

preparing a compaction by pressing a plurality

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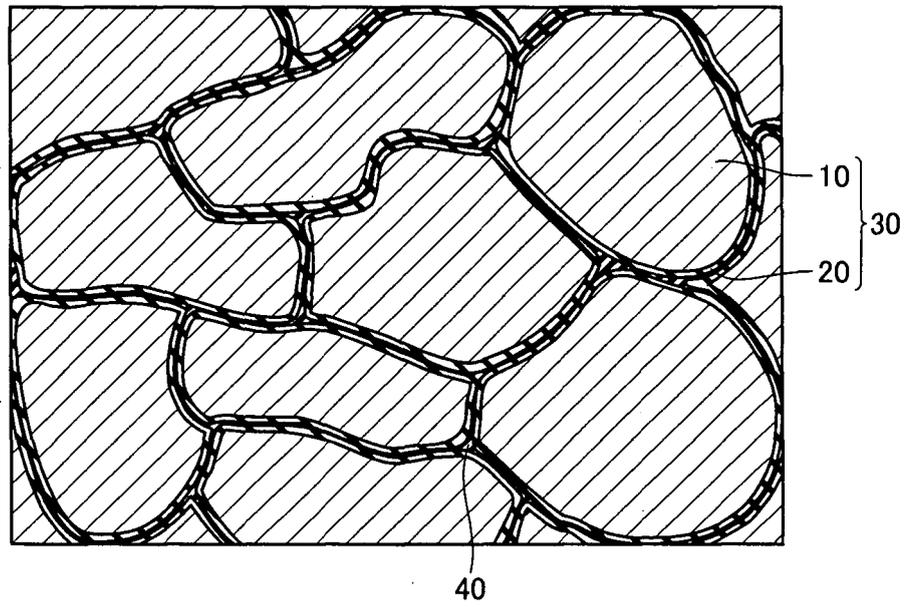
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FIG.1



## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2004/012846

A. CLASSIFICATION OF SUBJECT MATTER Int.Cl. <sup>7</sup> H01F1/33, H01F1/24, H01F41/02, B22F3/10		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) Int.Cl. <sup>7</sup> H01F1/33, H01F1/24, H01F41/02, B22F3/10		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Jitsuyo Shinan Koho 1922-1996 Toroku Jitsuyo Shinan Koho 1994-2004 Kokai Jitsuyo Shinan Koho 1971-2004 Jitsuyo Shinan Toroku Koho 1996-2004		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2003-37018 A (Daido Steel Co., Ltd.), 07 February, 2003 (07.02.03), Claims 1, 2; Par. Nos. [0020] to [0023] (Family: none)	1-3 4
Y	JP 8-67941 A (Sumitomo Special Metals Co., Ltd.), 12 March, 1996 (12.03.96), Par. No. [0006] (Family: none)	4
<input type="checkbox"/> Further documents are listed in the continuation of Box C.		<input type="checkbox"/> See patent family annex.
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