EUROPEAN PATENT SPECIFICATION

(54) POWDER COMPOSITION, METHOD FOR MAKING SOFT MAGNETIC COMPONENTS AND SOFT MAGNETIC COMPOSITE COMPONENT

POWDER COMPOSITION, METHOD FOR MAKING SOFT MAGNETIC COMPONENTS AND SOFT MAGNETIC COMPOSITE COMPONENT

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The present invention relates to iron-based powder compositions. More specifically, the invention concerns powder compositions for producing soft magnetic composite components by the powder metallurgical production route. The compositions facilitate the manufacture of the soft magnetic composite component having high density as well as valuable magnetic and mechanical properties.

BACKGROUND OF THE INVENTION

Soft magnetic materials are used for 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 Composite, SMC, materials are 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, the SMC parts are obtained. By using this powder metallurgical technique it is possible to produce materials giving a higher degree of freedom in the design of the SMC component than by using the steel laminates as the SMC material can carry a three dimensional magnetic flux and as three dimensional shapes can be obtained by the compaction process.

Two key characteristics of an iron core component are its magnetic permeability and core loss characteristics. The magnetic permeability of a material is an indication of its ability to become magnetised or its ability to carry a magnetic flux. Permeability is defined as the ratio of the induced magnetic flux to the magnetising force or field intensity. When a magnetic material is exposed to an alternating magnetic field, energy losses, core losses, occur due to both hysteresis losses and eddy current losses. The hysteresis loss is brought about by the necessary expenditure of energy to overcome the retained magnetic forces within the iron core component and is proportional to the frequency of the alternating field. The eddy current loss is brought about by the production of electric currents in the iron core component due to the changing flux caused by alternating current (AC) conditions and is proportional to the square of the frequency of the alternating field. A high electrical resistivity is then desirable in order to minimise the eddy currents and is of especial importance at higher frequencies. In order to decrease the hysteresis losses and to increase the magnetic permeability of a core component for AC applications it is generally desired to heat treat the compacted part.

Research in the powder metallurgical manufacture of magnetic core components using coated iron-based powders has been directed to the development of iron powder compositions that enhance certain physical and magnetic properties without detrimentally affecting other properties of the final component. Desired component properties include e.g. a high permeability through an extended frequency range, low core losses, high saturation induction, (high density) and high strength. Normally an increased density of the component enhances all of these properties.

The desired powder properties include suitability for compression moulding techniques, which i.a. means that the powder can be easily moulded into a high density, high strength component which can be easily ejected from the moulding equipment and that the components have smooth surface finish.

GB 682 897 discloses a process of preparing magnetic powder for magnetic cores having high resistivity and low power losses when operating at very high frequencies, which comprises impact milling a mixture of carbonyl iron powder, subject to excessive cluster formation, the particle size range of which does not substantially exceed a diameter of 5 micros, with a cluster-penetrating material which forms a solid, non-conductive, inert particle-separating deposit on the particles, the amount of the cluster-penetrating material being such as to yield the deposit in an amount from 0.2 to 5 % of the weight of the iron powder, and continuing impact milling of the mixture until the resulting powder has an apparent density when dry of at least 2.3 g/cm³, and the deposit is uniformly distributed throughout the mixture. The present invention concerns a new powder composition having the desired powder properties as well as the use of the powder composition for the preparation of soft magnetic composite components. The new composition can be compacted (and heat treated) to components having the desired properties.

The present invention also concerns a method for manufacturing soft magnetic iron-based components having excellent component properties.

SUMMARY OF THE INVENTION

In brief the powder composition according to the invention is made up by electrically insulated particles of a soft magnetic material and a fatty acid amide lubricant. Optionally a thermoplastic binder is present in the composition. The method according to the present invention includes mixing, compaction and optionally heat treatment of the obtained component resulting in a soft magnetic iron-based component having excellent properties.
The powder is a substantially pure, water atomised iron powder or a sponge iron powder having irregularly shaped particles. In this context the term "substantially pure" means that the powder should be substantially free from inclusions and that the amounts of the impurities O, C and N should be kept at a minimum. The average particle sizes are generally below 300 μm and above 10 μm. Examples of such powders are ABC 100.30, ASC 100.29, AT 40.29, ASC 200, ASC 300, NC 100.24, SC 100.26, MH 300, MH 40.28, MH 40.24 available from Hoganas AB, Sweden.

According to one embodiment of the invention the powders used have coarser particles than what is normal in common die pressing. In practice this means that the powders are essentially without fine particles. The term "essentially without fine particles" is intended to mean that less than about 10%, preferably less than 5% the powder particles have a size below 45 μm as measured by the method described in SS-EN 24 497. The average particle diameter is typically between 106 and 425 μm. The amount of particles above 212 μm is typically above 20%. The maximum particle size may be about 2 mm.

The size of the iron-based particles normally used within the PM industry is distributed according to a gaussian distribution curve with an average particle diameter in the region of 30 to 100 μm and about 10-30% of the particles are less than 45 μm. Thus, the powders used according to the present invention may have a particle size distribution deviating from that normally used. These coarse powders may be obtained by removing the finer fractions of the powder or by manufacturing a powder having the desired particle size distribution. The invention is however not limited to the coarse powders but also powders having the particle sizes normally used for die pressing within the PM industry are included in the present invention.

The electrical insulation of the powder particles may be made of an inorganic material. Especially suitable are the type of insulation disclosed in the US 634 8265, which concerns particles of a base powder consisting of essentially pure iron having an insulating oxygen- and phosphorus-containing barrier. As regards the coating it should be especially mentioned that the properties of the composite component may be influenced by the thickness of the coating. Powders having insulated particles are available as Somaloy™ 500 and 550 from Hoganas AB, Sweden.

The lubricant used according to the invention is selected from the group consisting of fatty acid amides. Particularly suitable amides are primary amides of saturated or unsaturated fatty acid having 12-24, preferably 14-22 C atoms and most preferably 18-22 C atoms. The lubricants are used in amounts of 0.05-2% and preferably less than 1.5% by weight of the composition. Especially preferred amounts of the lubricant are 0.05-1%, preferably 0.05-0.8 more preferably 0.1-0.8% and most preferably 0.1-0.5% by weight. Especially preferred lubricants are stearic acid amide, oleic acid amide, behenic acid amide, erucic acid amide, palmitic acid amide, the stearic acid amide being most preferred. In the US patent 6,537,389 stearic acid amide seemingly in combination with rapeseed oil methyl ester is mentioned as a lubricant in connection with a thermoplastic resin, polyphatalamide as a binder for the compaction of soft magnetic powders.

Solid lubricants generally have a density of about 1-2 g/cm³ which is very low in comparison to the density of the iron-based powder, which is about 7.8 g/cm³. As a consequence, inclusions of these less dense lubricants in the compositions will lower the theoretical density of the compacted component. It is therefore essential to keep the amount of lubricant at low levels in order to produce high-density components. However, low amounts of lubricants tend to give ejection problems. It has now unexpectedly been found that the type of lubricants mentioned above can be used in low amounts without ejection problems.

By replacing the internal lubricants, i.e. lubricants added to the iron-based powder mix, with lubrication of the die wall, DWL, in combination with high compaction pressures high green densities can be reached. One drawback with this known method when compacting insulated iron-based powder at high compaction pressures, is however that the insulation of the iron-based powder is easily damaged leading to high core losses at higher frequencies. Furthermore, the use of DWL will add further process complexity, it may prolong cycle times and decrease the production robustness in an industrial environment. According to the present invention the fatty acid amide may be used as the only additive to the insulated iron or iron-based powder, although for certain applications it is advantageous to add minor amounts of a thermoplastic resin, specifically polyphenylene sulfide (PPS). The term "minor amounts" should in this context be interpreted as less than 2, preferably less 0.8, more preferably less than 0.6 and most preferably less than 0.5% by weight of the composition. In amounts lower than 0.05 no effects of PPS have been observed. Specifically the amount of PPS could vary between 0.1 and 0.5 and preferably between 0.2 and 0.5 or 0.4% by weight. The addition of PPS is of particular interest when good frequency stability is required.

The combination of PPS and stearic acid is known from the patent application WO01/22448. The examples of this application disclose that a soft magnetic material can be produced by mixing an electrically insulated iron-based powder with PPS and stearic acid. The mixture is compacted at elevated temperature and the obtained compacted part is heat treated at 260°C in an atmosphere of nitrogen followed by a second heat treatment at 285 to 300°C. It has now unexpectedly been found that by using the new powder composition, which includes a fatty acid amide in stead of a
corresponding fatty acid several advantages can be obtained. Thus it has been found that the new powder has unex-
5 pectedly improved lubricating properties, which results in that lower ejection energy is needed to eject the compacted
part from the die, that higher densities and that better transverse rupture strength can be obtained. Furthermore, the
compaction step can be performed at ambient temperature. Also the heat treatment can be facilitated, as the first heat-
treating step, which is required according to the WO publication, can be omitted.

[0019] Iron-based magnetic powders, which have insulated particles and which are combined with thermoplastic resins,
10 are described in the US patent application 2002/0084440. In contrast to the particles according to the present invention
these previously known particles also include a rare earth element. Furthermore, the thermoplastic resin is used in
relatively large amounts, namely at least 5% by weight. Additionally, the particle size of the iron-based powder is quite
small (3 μm is mentioned as an example). A lubricant selected from a wide variety of chemical compounds may also be
included. These powder compositions are taught to be useful preferably for injection molding, extrusion, injection com-
pression molding and injection pressing for the preparation of highly weather-resistant bonded permanent magnets.

[0020] In order to prepare composite components according to the present invention the powder composition is first
15 uniaxially pressed in a die, which normally must not be lubricated, although the powder composition may also be used
in lubricated dies. The compacted component is then ejected from the die and optionally subjected to a heat treatment.

[0021] The compaction may be performed at ambient or elevated temperatures and at pressures up to 1500 MPa.
[0022] According to a preferred embodiment of the invention the compaction is performed in a moderately heated tool
as in this way not only the green density and the ejection behaviour but also the maximum relative permeability will be
improved. When comparing properties of components compacted at an elevated temperature and at a lower compaction
pressure to properties of components compacted to the same green density at ambient temperature and at a higher
compaction pressure the component compacted at an elevated temperature will have a higher permeability. For larger
components it may be necessary to elevate the temperature of the powder as well in order to achieve the improvements
according to the invention.

[0023] The heat treatment can be performed in one or several steps. A recommended one step heat treatment is
20 performed for a period of 30 minutes to 4 hours in an oxygen-containing atmosphere (air) at a temperature between 250
and 550°C.
[0024] Another alternative is to perform the heat treatment at 250-350°C for a period of 30 minutes to 3 hours in a air
or inert gas followed by a heat treatment for 15 minutes to 2 hours in an oxygen containing (air) atmosphere at a
temperature between 350 and 550°C.

[0025] A somewhat different heat treatment is recommended when PPS is included in the composition. Thus in this
case the heat treatment may be performed at 250-350°C for 30 minutes to 4 hours in an oxygen-containing atmosphere
(air). Another alternative is to perform the heat treatment at 250-350°C for 30 minutes to 3 hours in air or inert gas
followed by 300-500°C for 15 minutes to 2 hours in an oxygen containing atmosphere (air).

[0026] The possibility of performing the heat treatment by using different atmospheres, periods of time and temperatures
in order to obtain a final component having the desired properties makes the new powder composition especially attractive.
[0027] By compacting a composition comprising an iron- based insulated powder having coarse particles and a lubricant
as described above at high pressures, such as above 800 MPa, followed by heat treatment of the compacted component,
soft magnetic composite components having a density \( \geq 7.5 \text{ g/cm}^3 \), a maximum relative permeability, \( \mu_{\text{max}} \geq 600 \), a
coercive force, \( H_c \leq 250 \text{ A/m} \) and a specific resistivity, \( \rho \geq 20 \mu\Omega\text{m} \). Such components may be of interest for the demanding
applications required in e.g. stator and rotor components in electrical machines.

[0028] The invention is further illustrated by following examples.

EXAMPLE 1.

[0029] The following materials were used.
[0030] An iron-based, water atomized powder with particles having a thin inorganic coating (Somaloy™ 500, available
30 from Höganäs AB, Sweden) was used as starting material.
PPS powder,
Stearic acid powder, lubricant A.
Stearic acid amide powder, lubricant B

[0031] 3 kg of the base powder Somaloy™ 500 was mixed with PPS and stearic acid amide or stearic acid, according
to table 1.

Table 1. Powder mixes: Lubricants and PPS,(percent by weight)

<table>
<thead>
<tr>
<th>Sample number</th>
<th>PPS</th>
<th>Lubricant</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 1</td>
<td>0.60%</td>
<td>0.2% A</td>
</tr>
</tbody>
</table>
The powder mixes were compacted into ring samples with an inner diameter of 45 mm, outer diameter 55 mm and height 5 mm at 800 MPa at ambient (room) temperature. Ring samples with a height of 10 mm were also compacted and the ejection force was measured on these samples. The ejection energy is shown in Table 2. The results show that considerably lower ejection energy is obtained by using the fatty acid amide.

After compaction the parts were heat treated at 290°C for 120 minutes in air. The obtained heat-treated rings were wound with 25 turns. The relative AC inductance permeability was measured with an LCR-meter (HP4284A) according to standard IEC 60404-6, 2nd Edition 2003-06.

The drop in initial permeability (frequency stability) is shown in tables 3 and 4. The drop in initial permeability is expressed as the difference between the initial permeability at 10 and 100 kHz divided by the initial permeability at 10 kHz. Table 3 shows that by increasing the amount of the fatty acid amid from 0.3 to 0.5% a better frequency stability can be obtained. Table 4 shows that by using the fatty acid amid instead of the corresponding fatty acid a better frequency stability is obtained. Furthermore table 4 discloses that without PPS a larger drop in frequency stability is obtained. However the initial permeability at 1 kHz for A9 was found to be 95 compared with 75 for A3. A high initial permeability at lower frequencies is advantageous for some applications.

### Table 2. Ejection energy measured on ring samples with h=10 mm.

<table>
<thead>
<tr>
<th>Sample number</th>
<th>PPS</th>
<th>Lubricant</th>
<th>Ejection Energy (J/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 1</td>
<td>0.60%</td>
<td>0.2% A</td>
<td>52</td>
</tr>
<tr>
<td>A 2</td>
<td>0.50%</td>
<td>0.3% A</td>
<td>46</td>
</tr>
<tr>
<td>A 3</td>
<td>0.50%</td>
<td>0.3% B</td>
<td>38</td>
</tr>
<tr>
<td>A 4</td>
<td>0.30%</td>
<td>0.3% B</td>
<td>37</td>
</tr>
<tr>
<td>A 5</td>
<td>0.30%</td>
<td>0.4% B</td>
<td>33</td>
</tr>
<tr>
<td>A 6</td>
<td>0.30%</td>
<td>0.5% B</td>
<td>30</td>
</tr>
<tr>
<td>A 7</td>
<td>0.10%</td>
<td>0.3% B</td>
<td>41</td>
</tr>
<tr>
<td>A 8</td>
<td>0.20%</td>
<td>0.3% B</td>
<td>39</td>
</tr>
<tr>
<td>A 9</td>
<td>-</td>
<td>0.4% B</td>
<td>35</td>
</tr>
</tbody>
</table>

### Table 3, drop in initial permeability

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Dμ 10-100 kHz (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 4</td>
<td>7.4</td>
</tr>
<tr>
<td>A 5</td>
<td>5.2</td>
</tr>
<tr>
<td>A 6</td>
<td>4.2</td>
</tr>
</tbody>
</table>
The specific electrical resistivity was measured by a four point measuring method and is shown in table 5. From this table it can be concluded that by using the fatty acid amide in stead of the corresponding acid a considerably higher electrical resistivity can be obtained.

Samples were also tested with regard to Transverse Rupture Strength, TRS, after heat treatment at 290°C for 120 minutes in air. The TRS was tested according to ISO 3995. TRS was also tested on parts at a temperature of 200°C. The TRS is shown in Table 6. The sample with 0.5% PPS and 0.3% stearic acid amide (A 3) shows significantly higher TRS at both room temperature (RT) and 200°C compared with both the sample with 0.5% PPS and 0.3% stearic acid (A2) and the sample with 0.2% PPS + 0.6% stearic acid (A1). The density is higher for a mix with low total organic content, which will result in higher induction and permeability (μmax).

EXAMPLE 2.

The following materials were used.

PPS powder,
Stearic acid powder, lubricant A
Stearic acid amide powder, lubricant B
Behenic acid amide powder, lubricant C
Oleic acid amide powder, lubricant D
Kenolube™.

The base powder Somaloy™ 500 was mixed with PPS and lubricants according to the following table 7.
The powder mixes were compacted into test bars according to ISO 3995 at a compaction pressure of 800 MPa at ambient temperature. After compaction the parts were heat treated in a two-step heat treatment. The first step was performed at 290°C for 105 minutes in inert nitrogen atmosphere. This step was followed by a subsequent heat treatment step at 350°C for 60 minutes in air. Samples were tested with regard to Transverse Rupture Strength, TRS, according to ISO 3995.

Results from testing of transverse rupture strength are shown in table 8. As can be seen from table 8 samples prepared with mixtures including the fatty acid amide give sufficient TRS-values. A higher density after heat treatment is reached, which is beneficial in terms on induction and permeability. If the PPS content is reduced to 0.3% or less the TRS is increased to values above 80 MPa. The samples without PPS and with the stearic acid amide lubricant even have TRS values above 100 MPa. The use of Kenolube™, which is a conventionally used lubricant, does not result in the required transverse rupture strength.

Table 7. Powder mixes: Lubricants and PPS, percent by weight.

<table>
<thead>
<tr>
<th>Sample number</th>
<th>PPS</th>
<th>Lubricant</th>
</tr>
</thead>
<tbody>
<tr>
<td>B 1</td>
<td>0.50%</td>
<td>0.3% A</td>
</tr>
<tr>
<td>B 2</td>
<td>0.50%</td>
<td>0.3% B</td>
</tr>
<tr>
<td>B 3</td>
<td>0.50%</td>
<td>0.3% C</td>
</tr>
<tr>
<td>B 4</td>
<td>0.50%</td>
<td>0.3% D</td>
</tr>
<tr>
<td>B 5</td>
<td>0.30%</td>
<td>0.3% B</td>
</tr>
<tr>
<td>B 6</td>
<td>-</td>
<td>0.4% B</td>
</tr>
<tr>
<td>B 7</td>
<td>-</td>
<td>0.3% B</td>
</tr>
<tr>
<td>B 8</td>
<td>0.1%</td>
<td>0.3% B</td>
</tr>
<tr>
<td>B 9</td>
<td>0.2%</td>
<td>0.3% B</td>
</tr>
<tr>
<td>B 10</td>
<td>-</td>
<td>0.4% Kenolube™</td>
</tr>
</tbody>
</table>

Table 8. Density and TRS at room temperature

<table>
<thead>
<tr>
<th>Sample numbers</th>
<th>PPS</th>
<th>Lubricant</th>
<th>Density after HT</th>
<th>TRS-RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>B 1</td>
<td>0.50%</td>
<td>0.3% A</td>
<td>7.18</td>
<td>73</td>
</tr>
<tr>
<td>B 2</td>
<td>0.50%</td>
<td>0.3% B</td>
<td>7.22</td>
<td>68</td>
</tr>
<tr>
<td>B 3</td>
<td>0.50%</td>
<td>0.3% C</td>
<td>7.23</td>
<td>73</td>
</tr>
<tr>
<td>B 4</td>
<td>0.50%</td>
<td>0.3% D</td>
<td>7.24</td>
<td>74</td>
</tr>
<tr>
<td>B 5</td>
<td>0.30%</td>
<td>0.3% B</td>
<td>7.32</td>
<td>83</td>
</tr>
<tr>
<td>B 6</td>
<td>-</td>
<td>0.4% B</td>
<td>7.37</td>
<td>108</td>
</tr>
<tr>
<td>B 7</td>
<td>-</td>
<td>0.3% B</td>
<td>7.41</td>
<td>113</td>
</tr>
<tr>
<td>B 8</td>
<td>0.1%</td>
<td>0.3% B</td>
<td>7.35</td>
<td>88</td>
</tr>
<tr>
<td>B 9</td>
<td>0.2%</td>
<td>0.3% B</td>
<td>7.32</td>
<td>79</td>
</tr>
<tr>
<td>B 10</td>
<td>-</td>
<td>0.4% Kenolube™</td>
<td>7.42</td>
<td>32</td>
</tr>
</tbody>
</table>

EXAMPLE 3

This example shows that, in comparison with the commonly used Zinc Stearate and Ethylene bis stearamide lubricants, low ejection forces during ejection of compacted components and perfect surface finish of the ejected component are obtained, when the fatty acid amide lubricants according to the invention are used in low amount in combination with coarse powders and high compaction pressures.

Two kilos of a coarse soft magnetic iron-based powder, wherein the particles are surrounded by an inorganic
insulation according to US 6,348,265 were mixed with 0.2% by weight of lubricants according to table 9. The particle size distribution of the coarse iron-based powder is shown in table 10. Mix E and F are comparative examples containing known lubricants.

Table 9.

<table>
<thead>
<tr>
<th>Mix</th>
<th>Lubricant</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Behenamide</td>
</tr>
<tr>
<td>B</td>
<td>Erucamide</td>
</tr>
<tr>
<td>C</td>
<td>Stearamide</td>
</tr>
<tr>
<td>D</td>
<td>Oleylamide</td>
</tr>
<tr>
<td>E</td>
<td>Zinc Stearate</td>
</tr>
<tr>
<td>F</td>
<td>Ethylene bis stearamide</td>
</tr>
</tbody>
</table>

Table 10.

<table>
<thead>
<tr>
<th>Particle size (μm)</th>
<th>Weight %</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt;425</td>
<td>0.1</td>
</tr>
<tr>
<td>425-212</td>
<td>64.2</td>
</tr>
<tr>
<td>212-150</td>
<td>34.0</td>
</tr>
<tr>
<td>150-106</td>
<td>1.1</td>
</tr>
<tr>
<td>106-75</td>
<td>0.3</td>
</tr>
<tr>
<td>45-75</td>
<td>0.2</td>
</tr>
<tr>
<td>&lt;45</td>
<td>0</td>
</tr>
</tbody>
</table>

[0044] The obtained mixes were transferred to a die and compacted into cylindrical test samples (50 grams) with a diameter of 25 mm, in an uniaxially press movement at a compaction pressure of 1100 MPa. The used die material was conventional tool steel. During ejection of the compacted samples the ejection force was recorded. The total ejection energy/enveloping area needed in order to eject the samples was calculated. The following table 11 show ejection energy, green density and the surface finish.

Table 11

<table>
<thead>
<tr>
<th>Mix</th>
<th>Ejection energy (J/cm²)</th>
<th>Green density (g/cm³)</th>
<th>Surface finish</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>90</td>
<td>7.64</td>
<td>Perfect</td>
</tr>
<tr>
<td>B</td>
<td>83</td>
<td>7.65</td>
<td>Perfect</td>
</tr>
<tr>
<td>C</td>
<td>93</td>
<td>7.63</td>
<td>Perfect</td>
</tr>
<tr>
<td>D</td>
<td>70</td>
<td>7.67</td>
<td>Acceptable</td>
</tr>
<tr>
<td>E</td>
<td>117</td>
<td>7.66</td>
<td>Not Acceptable</td>
</tr>
<tr>
<td>F</td>
<td>113</td>
<td>7.64</td>
<td>Perfect</td>
</tr>
</tbody>
</table>

EXAMPLE 4

[0045] The following example illustrates the effect of the particle size distribution of the soft magnetic iron-based powder on ejection behaviour and green density. A “coarse” powder according to example 3 was used. The particle size distribution of the “fine” powder is given in table 12. The mixes were prepared using 0.2% stearamide by weight according to the procedure in example 3. The mixture based on the “fine” powder is marked sample H and were compared with sample C.
The mixes were compacted into cylindrical samples according to the procedure used in example 3. The following table 13 shows green density and the surface appearance.

<table>
<thead>
<tr>
<th>Mix</th>
<th>Green density (g/cm³)</th>
<th>Surface finish</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>7.63</td>
<td>Perfect</td>
</tr>
<tr>
<td>H</td>
<td>7.53</td>
<td>Acceptable</td>
</tr>
</tbody>
</table>

As can be seen from table 13 the composition containing fine powder results in a lower green density and deteriorated surface finish.

EXAMPLE 5.

This example compares a known lubricant, ethylene bisstearamide (EBS), and an example of the lubricant stearamide. A “coarse” powder according to example 3 was used was mixed with EBS and stearamide, respectively, according to table 14. The samples were prepared according to the procedure in example 3.

<table>
<thead>
<tr>
<th>Mix</th>
<th>EBS (weight%)</th>
<th>Stearamide (weight%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.20</td>
<td>--</td>
</tr>
<tr>
<td>2</td>
<td>0.30</td>
<td>--</td>
</tr>
<tr>
<td>3</td>
<td>0.40</td>
<td>--</td>
</tr>
<tr>
<td>4</td>
<td>0.50</td>
<td>--</td>
</tr>
<tr>
<td>5</td>
<td>--</td>
<td>0.10</td>
</tr>
<tr>
<td>6</td>
<td>--</td>
<td>0.20</td>
</tr>
<tr>
<td>7</td>
<td>--</td>
<td>0.30</td>
</tr>
</tbody>
</table>

The powder mixes were compacted into rings with an inner diameter of 45 mm, an outer diameter of 55 mm and the height 10 mm at 1100 MPa. During ejection of the compacted samples, the total ejection energy/enveloping area needed in order to eject the samples from the die was calculated. The following table 15 shows the calculated ejection energy/area, green density and the surface appearance.

<table>
<thead>
<tr>
<th>Mix</th>
<th>Ejection energy [J/cm²]</th>
<th>Density [g/cm³]</th>
<th>Surface appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>54</td>
<td>7.65</td>
<td>Not acceptable</td>
</tr>
</tbody>
</table>
As can be seen from table 15 the new lubricant can be added in amount as low as 0.2% and still a perfect surface finish can be obtained whereas the for the reference lubricant, EBS, the lowest addition is 0.4% for obtaining a perfect surface finish.

EXAMPLE 6

This example compares the magnetic properties of components manufactured with a minimum amount of the lubricating components stearamide and EBS respectively, in order to achieve similar values of ejection energy. Components made from mix 2 and mix 6 according to example 5 were compared regarding magnetic properties after heat treatment.

Ring samples according to example 5 except that the height were 5 mm were compacted. The green samples were heat treated at 300°C for 60 minutes in air followed by a second step of heat treatment at 530°C for 30 minutes in air. The obtained heat-treated rings were wounded with 100 sense and 100 drive turns and tested in a Brockhaus hysteresisgraph. The following table 16 shows the induction level at 10 kA/m, maximum relative permeability, coercive force $H_c$ and core loss at 400 Hz, 1T.

As can bee seen in table 16 the soft magnetic properties are superior for components according to the present invention.

EXAMPLE 7

The following example shows the influence of die temperature on the ejection properties and green density of compacted samples. In this example the primary amide, stearamide, was selected as the amide lubricant according to the invention. 0.2% of stearamide was added to 2 kg of a coarse soft magnetic electrically insulated iron-based powder according to the procedure of example 3.

The powder mixes were compacted into rings having an inner diameter of 45 mm, an outer diameter of 55 mm and a height of 10 mm, at a compaction pressure of 1100 MPa. During ejection of the compacted samples the ejection forces were recorded. The total ejection energy/enveloping area needed in order to eject the samples from the die was calculated. The following table 17 shows ejection energy, green density and the surface appearance of the samples compacted at different temperature of the die.
As can be seen from table 17 the ejection energy and the green density is positively influenced by increasing die temperature.

EXAMPLE 8

This example compares component properties of components manufactured according to the present invention to properties of components compacted with the aid of DWL. In both the inventive example and the comparative example a "coarse" powder according to example 3 was used. As lubricant in the inventive example 0.2% by weight of stearamide was used and the obtained powder composition was compacted at a controlled die temperature of 80°C into ring samples having a green density of 7.6 g/cm³. In the comparative example no internal lubricant was used, instead DWL was applied. Ring samples were compacted to a density of 7.6 g/cm³ at ambient temperature.

The ring samples outer diameter was 55 mm, inner diameter 45 mm and height 5 mm.

After compaction heat-treatment was done according to table 18. The specific electrical resistivity was measured by a 4-point method. Prior to magnetic measurements in the hysteresis graph the ring samples were wound with 100 drive and 100 sense turns. The DC properties were acquired from a loop at 10kA/m. The core loss was measured at different frequencies at 1T. In figure 1 the core loss/cycle is plotted as a function of frequency.

From the table 18 and figure 1 it can be concluded that the present invention gives significantly lower core loss in alternating fields due to lower $H_c$ and higher resistivity compared to the DWL-method.

EXAMPLE 9

In this example it is shown that iron-powder cores with excellent magnetic properties can obtained by the present invention. The positive effect of elevated die temperature on the maximal relative permeability is also shown.

A "coarse" powder according to example 3 was mixed with various contents and types of lubricants. Both ring samples (OD=55, ID=45, h=5mm) and bars (30x12x6 mm) were manufactured with the process conditions given in table 19.

The density was determined by measuring the mass and dimensions of the ring samples. The specific electrical resistivity was measured by a 4-point method on the ring samples. Prior to magnetic measurements in a Brockhaus hysteresisgraph the ring samples were wound with 100 drive and 100 sense turns. The DC-properties such as $P_{max}$ and $H_c$ were acquired from a loop at 10kA/m while the core loss was measured at 1T and 400Hz. The transverse rupture strength (TRS) of the heat-treated parts was determined on the test bars by a three-point bending method.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat-treatment</th>
<th>$B_{10kA/m}$</th>
<th>$H_c$ [A/m]</th>
<th>$\rho$ [$\mu\Omega m$]</th>
<th>Core loss @1 T, 400Hz [W/kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present invention</td>
<td>530°C, 30min air</td>
<td>1.65</td>
<td>192</td>
<td>103</td>
<td>41</td>
</tr>
<tr>
<td>DWL- method</td>
<td>none</td>
<td>1.66</td>
<td>305</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>DWL- method</td>
<td>530°C, 30min air</td>
<td>1.66</td>
<td>189</td>
<td>3</td>
<td>109</td>
</tr>
</tbody>
</table>
Table 19: Process conditions for ring samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Type of lubricant</th>
<th>Amount Lubricant (%wt)</th>
<th>Compacting pressure (MPa)</th>
<th>Die temperature (°C)</th>
<th>Heat treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Stearamide</td>
<td>0.2</td>
<td>1100</td>
<td>25</td>
<td>300°C 45 min, air + 520°C*, air</td>
</tr>
<tr>
<td>2</td>
<td>Stearamide</td>
<td>0.2</td>
<td>1100</td>
<td>80</td>
<td>300°C 45 min, air + 520°C*, air</td>
</tr>
<tr>
<td>3</td>
<td>Stearamide</td>
<td>0.2</td>
<td>800</td>
<td>80</td>
<td>530°C, 30 min, air</td>
</tr>
<tr>
<td>4</td>
<td>Stearamide</td>
<td>0.2</td>
<td>1100</td>
<td>25</td>
<td>530°C, 30 min, air</td>
</tr>
<tr>
<td>5</td>
<td>Stearamide</td>
<td>0.2</td>
<td>1100</td>
<td>80</td>
<td>530°C, 30 min, air</td>
</tr>
<tr>
<td>6</td>
<td>Stearamide</td>
<td>0.1</td>
<td>1100</td>
<td>85</td>
<td>530°C, 30 min, air</td>
</tr>
<tr>
<td>7</td>
<td>Stearamide</td>
<td>0.3</td>
<td>800</td>
<td>25</td>
<td>300°C, 1h, air + 530°C, 30 min, air</td>
</tr>
<tr>
<td>8</td>
<td>Stearamide</td>
<td>0.3</td>
<td>800</td>
<td>80</td>
<td>300°C, 1h, air + 530°C, 30 min, air</td>
</tr>
<tr>
<td>9</td>
<td>Stearamide</td>
<td>0.3</td>
<td>1100</td>
<td>25</td>
<td>300°C, 1h, air + 530°C, 30 min, air</td>
</tr>
<tr>
<td>10</td>
<td>Stearamide</td>
<td>0.3</td>
<td>1100</td>
<td>80</td>
<td>300°C, 1h, air + 530°C, 30 min, air</td>
</tr>
<tr>
<td>11</td>
<td>Erucamide</td>
<td>0.2</td>
<td>1100</td>
<td>25</td>
<td>330°C, 2h, air + 530°C, 30 min, air</td>
</tr>
<tr>
<td>12</td>
<td>Erucamide</td>
<td>0.2</td>
<td>1100</td>
<td>25</td>
<td>340°C, 2h, N₂ + 530°C, 30 min, air</td>
</tr>
</tbody>
</table>

*increasing temperature approx 4°C/min in the component up to 520°C

Table 20: Measurements of component properties

<table>
<thead>
<tr>
<th>Sample</th>
<th>Density (g/cm³)</th>
<th>$\mu_{\text{max}}$</th>
<th>$H_c$ (A/m)</th>
<th>Resistivity ($\mu\text{Ohm}\cdot\text{m}$)</th>
<th>Core loss at 1T 400 Hz (W/kg)</th>
<th>TRS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.62</td>
<td>754</td>
<td>209</td>
<td>473</td>
<td>42</td>
<td>93</td>
</tr>
<tr>
<td>2</td>
<td>7.63</td>
<td>852</td>
<td>204</td>
<td>230</td>
<td>40</td>
<td>97</td>
</tr>
<tr>
<td>3</td>
<td>7.60</td>
<td>718</td>
<td>208</td>
<td>103</td>
<td>43</td>
<td>n.a</td>
</tr>
<tr>
<td>4</td>
<td>7.62</td>
<td>602</td>
<td>198</td>
<td>591</td>
<td>39</td>
<td>59</td>
</tr>
<tr>
<td>5</td>
<td>7.65</td>
<td>861</td>
<td>178</td>
<td>98</td>
<td>37</td>
<td>68</td>
</tr>
<tr>
<td>6</td>
<td>7.71</td>
<td>918</td>
<td>177</td>
<td>66</td>
<td>38</td>
<td>78</td>
</tr>
<tr>
<td>7</td>
<td>7.49</td>
<td>669</td>
<td>228</td>
<td>574</td>
<td>46</td>
<td>70</td>
</tr>
<tr>
<td>8</td>
<td>7.53</td>
<td>880</td>
<td>202</td>
<td>33</td>
<td>48</td>
<td>81</td>
</tr>
<tr>
<td>9</td>
<td>7.56</td>
<td>672</td>
<td>224</td>
<td>515</td>
<td>44</td>
<td>67</td>
</tr>
<tr>
<td>10</td>
<td>7.62</td>
<td>860</td>
<td>203</td>
<td>64</td>
<td>43</td>
<td>76</td>
</tr>
<tr>
<td>11</td>
<td>7.62</td>
<td>633</td>
<td>192</td>
<td>414</td>
<td>38</td>
<td>54</td>
</tr>
<tr>
<td>12</td>
<td>7.68</td>
<td>738</td>
<td>205</td>
<td>614</td>
<td>39</td>
<td>67</td>
</tr>
</tbody>
</table>
Claims

1. Powder composition consisting of irregularly shaped particles of a soft magnetic material of substantially pure water-atomized iron or sponge iron powder, wherein less than 10% by weight of said powder particles have a particle size less than 45 μm, and wherein said powder particles being provided with an electrically insulating layer, 0.05-2% by weight of a lubricant selected from the group consisting of primary amides of saturated or unsaturated, straight fatty acids having 12-24 C atoms, and optionally polyphenylene sulfide in a concentration less than 2% by weight, wherein said powder composition is capable of producing by uniaxial compaction a soft magnetic component.

2. Composition according to claim 1 wherein the fatty acid has 14-22 C atoms.

3. Composition according to claim 1 or 2, wherein the fatty acid amide is selected from the group consisting of stearic acid amide, oleic acid amide, behenic acid amide, erucic acid amide, palmitic acid amide.

4. Composition according to any one of the claims further including said polyphenylene sulfide.

5. Composition according to claim 4, wherein the poly phenylene sulfid is used in an amount of 0.05-2.0% by weight.

6. Composition according to any one of the claims 1-5, wherein the fatty acid amide is present in an amount of 0.05-1, preferably 0.05-0.8, more preferably 0.1-0.8, most preferably 0.1-0.5% by weight.

7. Composition according to any one of the claims 1-6, wherein the electrically insulating layer is made up of an inorganic material.

8. Composition according to claim 7, wherein the layer includes oxygen and phosphorus.

9. Composition according to any one of the previous claims, wherein less than 5% by weight of said powder particles have a particle size less than 45 μm.

10. Composition according to claim 9, wherein at least 20% of the particles have a particle size above 212 μm.

11. A method for making soft magnetic components comprising the steps of:

   a) mixing irregularly shaped particles of a soft magnetic substantially pure water-atomized iron or sponge iron powder, wherein less than 10 by weight of said powder particles have a particle size less than 45 μm, and wherein the particles are surrounded by an electrically insulating layer, and 0.05-2% by weight of a lubricant selected from the group consisting of primary amides of saturated or unsaturated, straight fatty acid having 12-24 C atoms, and optionally polyphenylene sulfide in a concentration less than 2% by weight,
   b) uniaxially compacting the resulting composition, and
   c) optionally subjecting the obtained component to heat treatment.

12. A method according to claim 11 wherein the compaction is performed at an elevated temperature.

13. A method according to claim 11 or 12 wherein the amount of lubricant is between 0.05-0.8%, preferably 0.1-0.8, and more preferably 0.1-0.5% by weight.

14. A method according to any one of the claims 11-13, wherein the compaction is performed at a compaction pressure above 800 MPa.

15. A method according to any of claims 11-14 wherein less than 5% of said powder particles have a particle size less than 45 μm.

16. A method according to any of claims 11-15 wherein the heat treatment is performed between 250°C and 550 °C.

17. A method according to any one of claims 11-15 wherein the heat treatment is performed in a first step up to 350° followed by heat treatment up to 550°C.

18. A method according to any one of claims 11-17
wherein the heat treatment is performed in air or inert atmosphere.

**Patentansprüche**

1. Pulverzusammensetzung, die aus unregelmäßig geformten Teilchen eines weichmagnetischen Materials aus im Wesentlichen reinem, mit Wasser zerstäubtem Eisen- oder Eisenschwamppulver besteht, wobei weniger als 10 Gew.-% der Pulverteilchen eine Teilchengröße von weniger als 45 µm aufweisen, und wobei die Pulverteilchen mit einer elektrisch isolierenden Schicht versehen sind, 0,05 bis 2 Gew.-% eines Schmiermittels, das ausgewählt ist aus der Gruppe, bestehend aus primären Amiden gesättigter oder ungesättigter, gerader Fettsäuren mit 12 bis 24 C-Atomen und gegebenenfalls Polyphenylensulfid in einer Konzentration von weniger als 2 Gew.-%, wobei die Pulverzusammensetzung in der Lage ist, durch uniaxiale Verdichtung eine weichmagnetische Komponente herzustellen.

2. Zusammensetzung nach Anspruch 1, wobei die Fettsäure 14 bis 22 C-Atome aufweist.

3. Zusammensetzung nach Anspruch 1 oder 2, wobei das Fettsäureamid ausgewählt ist aus der Gruppe, bestehend aus Stearinsäureamid, Oleinsäureamid, Behensäureamid, Eurinsäureamid, Palmitinsäureamid.


5. Zusammensetzung nach Anspruch 4, wobei das Polyphenylensulfid in einer Menge von 0,05 bis 2,0 Gew.-% verwendet wird.

6. Zusammensetzung nach einem der Ansprüche 1 bis 5, wobei das Fettsäureamid in einer Menge von 0,05 bis 1, vorzugsweise von 0,05 bis 0,8, besonders bevorzugt von 0,1 bis 0,8, am meisten bevorzugt von 0,1 bis 0,5 Gew.-%, vorliegt.

7. Zusammensetzung nach einem der Ansprüche 1 bis 6, wobei die elektrisch isolierende Schicht aus einem anorganischen Material zusammengesetzt ist.

8. Zusammensetzung nach Anspruch 7, wobei die Schicht Sauerstoff und Phosphor umfasst.


10. Zusammensetzung nach Anspruch 9, wobei mindestens 20 % der Teilchen eine Teilchengröße oberhalb von 212 µm aufweisen.

11. Verfahren zur Herstellung weichmagnetischer Komponenten, umfassend die Schritte:

   a) Mischen unregelmäßig geformter Teilchen eines weichmagnetischen, im Wesentlichen reinem, mit Wasser zerstäubtem Eisen- oder Eisenschwamppulver, wobei weniger als 10 Gew.-% der Pulverteilchen eine Teilchengröße von weniger als 45 µm aufweisen, und wobei die Teilchen mit einer elektrisch isolierenden Schicht umgeben sind, und 0,05 bis 2 Gew.-% eines Schmiermittels, das ausgewählt ist aus der Gruppe, bestehend aus primären Amiden gesättigter oder ungesättigter, gerader Fettsäuren mit 12 bis 24 C-Atomen und gegebenenfalls Polyphenylensulfid in einer Konzentration von weniger als 2 Gew.-%,

   b) uniaxiales Verdichten der sich ergebenden Zusammensetzung, und

   c) gegebenenfalls Unterziehen der erhaltenen Komponente einer Wärmebehandlung.

12. Verfahren nach Anspruch 11, wobei die Verdichtung bei einer erhöhten Temperatur durchgeführt wird.

13. Verfahren nach Anspruch 11 oder 12, wobei die Menge des Schmiermittels zwischen 0,05 bis 0,8 %, vorzugsweise 0,1 bis 0,8 und besonders bevorzugt 0,1 bis 0,5 Gew.-% beträgt.

14. Verfahren nach einem der Ansprüche 11 bis 13, wobei die Verdichtung bei einem Verdichtungsdruck oberhalb von 800 MPa durchgeführt wird.
Verfahren nach einem der Ansprüche 11 bis 14, wobei weniger als 5 Gew.-% der Pulverteilchen eine Teilchengröße von weniger als 45 \( \mu \text{m} \) aufweisen.

Verfahren nach einem der Ansprüche 11 bis 15, wobei die Wärmebehandlung zwischen 250 °C und 550 °C durchgeführt wird.

Verfahren nach einem der Ansprüche 11 bis 15, wobei die Wärmebehandlung in einem ersten Schritt bis zu 350 °C durchgeführt wird, gefolgt von einer Wärmebehandlung bis zu 550 °C.

Verfahren nach einem der Ansprüche 11 bis 17, wobei die Wärmebehandlung in Luft oder einer inerten Atmosphäre durchgeführt wird.

Revendications

1. Composition de poudre constituée de particules de forme irrégulière d’un matériau à aimantation temporaire de poudre de fer ou de fer spongieux atomisée à l’eau et sensiblement pure, dans laquelle moins de 10 % en poids desdites particules de poudre ont une taille de particule inférieure à 45 \( \mu \text{m} \), et dans laquelle lesdites particules de poudre sont dotées d’une couche électriquement isolante, de 0,05 à 2 % en poids d’un lubrifiant sélectionné dans le groupe constitué d’amides primaires d’acides gras linéaires, saturés ou insaturés, ayant 12 à 24 atomes de carbone, et facultativement de sulfure de polyphénylène en une concentration inférieure à 2 % en poids, dans laquelle ladite composition de poudre est capable de produire par compactage uniaxial un composant à aimantation temporaire.

2. Composition selon la revendication 1, dans laquelle l’acide gras a 14 à 22 atomes de carbone.


5. Composition selon la revendication 4, dans laquelle le sulfure de polyphénylène est utilisé en une quantité de 0,05 à 2,0 % en poids.

6. Composition selon l’une quelconque des revendications 1 à 5, dans laquelle l’amide d’acide gras est présent en une quantité de 0,05 à 1, de préférence de 0,05 à 0,8, de manière davantage préférée de 0,1 à 0,8, idéalement de 0,1 à 0,5 % en poids.

7. Composition selon l’une quelconque des revendications 1 à 6, dans laquelle la couche électriquement isolante est constituée d’un matériau inorganique.

8. Composition selon la revendication 7, dans laquelle la couche comprend de l’oxygène et du phosphore.

9. Composition selon l’une quelconque des revendications précédentes, dans laquelle moins de 5 % en poids desdites particules de poudre ont une taille de particule inférieure à 45 \( \mu \text{m} \).

10. Composition selon la revendication 9, dans laquelle au moins 20 % des particules ont une taille de particule supérieure à 212 \( \mu \text{m} \).

11. Procédé de fabrication de composants à aimantation temporaire qui comprend les étapes consistant à :

   a) mélanger des particules de forme irrégulière d’une poudre de fer ou de fer spongieux atomisée à l’eau et sensiblement pure à aimantation temporaire, dans laquelle moins de 10 % en poids desdites particules de poudre ont une taille de particule inférieure à 45 \( \mu \text{m} \), et

   dans laquelle les particules sont entourées par une couche électriquement isolante, et de 0,05 à 2 % en poids d’un lubrifiant sélectionné dans le groupe constitué d’amides primaires d’un acide gras linéaire, saturé ou insaturé, ayant 12 à 24 atomes de carbone, et facultativement de sulfure de polyphénylène en une concentration
inférieure à 2 % en poids,
b) compacter uniaxialement la composition obtenue, et
c) soumettre facultativement le composant obtenu à un traitement thermique.

12. Procédé selon la revendication 11, dans lequel le compactage est réalisé à une température élevée.

13. Procédé selon la revendication 11 ou 12, dans lequel la quantité de lubrifiant est comprise entre 0,05 et 0,8 %, de préférence entre 0,1 et 0,8, et de manière davantage préférée entre 0,1 et 0,5 % en poids.

14. Procédé selon l’une quelconque des revendications 11 à 13, dans lequel le compactage est réalisé à une pression de compactage supérieure à 800 MPa.

15. Procédé selon l’une quelconque des revendications 11 à 14, dans lequel moins de 5 % desdites particules de poudre ont une taille de particule inférieure à 45 μm.

16. Procédé selon l’une quelconque des revendications 11 à 15, dans lequel le traitement thermique est réalisé entre 250 °C et 550 °C.

17. Procédé selon l’une quelconque des revendications 11 à 15, dans lequel le traitement thermique est réalisé dans une première étape jusqu’à 350 °C, suivi par un traitement thermique jusqu’à 550 °C.

18. Procédé selon l’une quelconque des revendications 11 à 17, dans lequel le traitement thermique est réalisé à l’air ou dans une atmosphère inerte.
Figure 1: Core loss/cycle (loop) at 1 T as a function of frequency
REFERENCES CITED IN THE DESCRIPTION

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