The present invention relates to a process of improving the electric and magnetic properties of pulverulent metals and alloys, especially of nickel and iron and nickel-iron alloys.

According to the process of U. S. Patent No. 2,941,493, pulverulent alloys, in particular nickel-iron alloys, are obtained in an especially advantageous manner by thermally decomposing a mixture of the carbonyls of the desired alloy constituents, coating the particles of the resulting metal powder with a "substance hindering the fritting together thereof" and then heating the coated powder in a non-oxidizing, i.e. an inert or reducing atmosphere so that alloy formation takes place by diffusion within the particles of the metal powder without smelting.

I have now found that pulverulent metallic substances with valuable electric and magnetic properties can be obtained by coating the metallic powder particles with inorganic halides which are practically non volatile at high temperatures, heating the coated powder in a non-oxidizing atmosphere and removing the said halides from the heated particles to a far-reaching degree. Especially pulverulent alloys which are obtained by thermal decomposition of mixtures of metal carbonyls, such as nickel-iron powder, can be materially improved by practicing the thermal treatment as described in said U. S. Patent No. 2,941,493, in the presence of inorganic halides which are not volatile or but little so at the temperatures employed for the said thermal treatment. Also the properties of other nickel-iron powders, for instance of a powder obtained by evaporating an aqueous solution containing the nitrates of nickel and iron, glowing and reducing the residue and comminuting the mass, can be considerably improved by annealing the obtained powder in the presence of halides of the above-mentioned nature. The same is true with finely divided iron which has been prepared by comminuting iron pieces to powder form.

Water soluble inorganic halides, as for example sodium chloride, barium chloride or other alkaline metal or alkaline earth metal halides, are very suitable for the said purpose, since they may be readily removed and recovered without losses from the powder after the thermal treatment, by lixiviation with water. The metallic powder thus obtained can be worked into mass cores in known manner together with an insulation material most suitable for the purpose. In some cases the lixiviation may be advantageously carried out by means of organic solvents, such as alcohol, for example if the metallic powder is easily oxidized by water. It is not necessary to completely remove the halides by lixiviation; an unremoved portion of it acts as an insulation means in the manufacture of the cores.

The amount of halides to be added to the powder may vary within wide limits, but should be in all cases so calculated as to prevent the powder from sintering. It is preferable to use from 1 to 30 parts by weight of a halide to 100 parts by weight of the metallic powder.

The addition substances are preferably mixed as intimately as possible with the powder, for example by grinding them in a ball mill or making them into a paste by means of a solution or a suspension of the addition substance.

It offers no difficulty to incorporate the powder with further metal components by adding to the mixture a readily reducible metal compound, for example a copper or cobalt compound, as the chlorides of the said metals, and glowing the mass in a reducing atmosphere.

The following examples will further illustrate how the present invention may be carried out in practice, but the invention is not restricted to the said examples.

Example 1

A nickel-iron powder containing 50 per cent of nickel and prepared by thermal decomposition of a mixture of nickel carbonyl and iron carbonyl, was worked up into mass cores firstly without pretreatment (core No. 1), secondly after a previously proposed treatment (core No. 2) and thirdly after a treatment according to the present invention (core No. 3). A mixture of waterglass and kaolin was used in each case as the
insulation medium. The data of the electric and magnetic properties of these cores are the follow-

<table>
<thead>
<tr>
<th>Core No.</th>
<th>Initial permeability μ</th>
<th>Eddy current α</th>
<th>Hysteresis h</th>
<th>After-effect losses η</th>
<th>Hysteresis factor $h/\sqrt{\mu}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>27.3</td>
<td>4.0</td>
<td>28.2</td>
<td>18.1</td>
<td>6.43</td>
</tr>
<tr>
<td>2</td>
<td>72.5</td>
<td>48.8</td>
<td>33.8</td>
<td>10.0</td>
<td>6.27</td>
</tr>
<tr>
<td>3</td>
<td>94.7</td>
<td>18.0</td>
<td>42.4</td>
<td>0.45</td>
<td>6.87</td>
</tr>
</tbody>
</table>

In the pretreatment for core No. 2, the nickel-iron powder was made into a paste with 5 per cent of aluminum hydroxide sol and heated at 1000° C. for 6 hours in a hydrogen atmosphere. The lumps formed were reduced to a fine powder by grinding in a ball mill for 24 hours, of which powder 10 per cent passed through a sieve with 10,000 meshes per square centimeter. The sifted portion was heated at 700° C. for 3 hours in a hydrogen current, again ground for 6 hours and passed through a sieve with 10,000 meshes per square millimeter.

According to the present invention (core No. 3), the nickel-iron powder was finely ground with 30 per cent of its weight of sodium chloride in a ball mill and heated at 1000° C. for 4 hours in a hydrogen current. After 30 minutes' grinding the product had already been reduced to sufficient fineness. After dissolving out the sodium chloride by means of water the powder was heated again at 800° C. for 5 hours in a hydrogen current, ground for a short time and passed through a sieve with 10,000 meshes per square centimeter.

Example 2

A nickel-iron powder containing 50 per cent of nickel was prepared by evaporating an aqueous solution of nickel nitrate and iron nitrate (in equal proportions as to metal content), blowing the residue and reducing the resulting oxides with hydrogen, and passed through a sieve with 16,900 meshes per square centimeter. The powder so obtained was worked up into mass cores firstly without pretreatment (core No. 4), secondly after a previously proposed treatment (core No. 5) and thirdly after a treatment according to the present invention (core No. 6). A mixture of water-glass and kaolin was used in each case as insulating medium. In the pretreatment for core No. 5 the nickel-iron powder was made into a paste by means of 5 per cent of aluminum hydroxide sol and glazed at 950° C. for 6 hours in a hydrogen atmosphere. The glazed mass was ground, again heated for 4 hours at 900° C. in a hydrogen atmosphere and passed through a sieve with 16,900 meshes per square centimeter.

According to the present invention (core No. 3) the nickel-iron powder was mixed with 30 per cent of barium chloride, heated at 950° C. for 6 hours in a hydrogen atmosphere and ground. After dissolving out the barium chloride with water, the powder was again heated at 900° C. for 4 hours in a hydrogen atmosphere and sifted through a sieve with 16,900 meshes per square centimeter.

By comparison of the three cores the following data were found:

<table>
<thead>
<tr>
<th>Core No.</th>
<th>Initial permeability μ</th>
<th>Eddy current α</th>
<th>Hysteresis h</th>
<th>After-effect losses η</th>
<th>Hysteresis factor $h/\sqrt{\mu}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>36.9</td>
<td>18.0</td>
<td>45.0</td>
<td>4.1</td>
<td>7.45</td>
</tr>
<tr>
<td>2</td>
<td>40.4</td>
<td>1.98</td>
<td>40.8</td>
<td>1.57</td>
<td>8.52</td>
</tr>
<tr>
<td>3</td>
<td>43.1</td>
<td>0.98</td>
<td>27.7</td>
<td>11.7</td>
<td>8.74</td>
</tr>
</tbody>
</table>

Example 3

An iron powder obtained by grinding compact iron pieces is divided into two portions. Portion 1 is made into a paste with 5 per cent of aluminum hydroxide sol, heated at 850° C. for 6 hours in a hydrogen atmosphere, ground, again glazed at 800° C. for 6 hours and passed through a sieve with 10,000 meshes per square centimeter (core No. 7).

Portion 2 was mixed with 30 per cent of its weight of barium chloride and heated at 850° C. for 6 hours in a hydrogen current. After grinding and dissolving out the barium chloride, the powder was again glazed at 800° C. for 6 hours and passed through a sieve with 10,000 meshes per square centimeter (core No. 8).

By comparison of both cores it was found that core No. 7 had a permeability of 63.1 and a hysteresis factor $h/\sqrt{\mu} = 45.9$

whereas core No. 8 had a permeability of 76.8 and a hysteresis factor $h/\sqrt{\mu} = 19.2$

What I claim is:

1. A process of producing pulverulent nickel-iron alloys for electromagnetic purposes which comprises thermally decomposing a mixture of nickel carbonyl and iron carbonyl, coating the metal particles of the resulting powder with inorganic halides which are practically not volatile at high temperatures and are selected from the class consisting of alkaline metal and alkaline earth metal halides, heating the coated powder in a non-oxidizing atmosphere to a temperature of from about 500 to about 1000° C. and removing the greater part of said halides from the heated particles.

2. The process as defined in claim 1 wherein said inorganic halide is an alkaline earth metal halide.

3. The process as defined in claim 1 wherein said inorganic halide is an alkaline earth metal halide.

4. The process of producing pulverulent nickel-iron alloys for electromagnetic purposes which comprises thermally decomposing a mixture of nickel carbonyl and iron carbonyl, coating the metal particles of the resulting powder with sodium chloride, heating the coated powder to 1000° C. in a current of hydrogen, grinding the resulting product, dissolving out the sodium chloride with water to remove the greater part thereof, and reheating the product to 800° C. in a current of hydrogen.

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