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FINELY DIVIDED MAGNETIC SUBSTANCE

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The present invention relates to the production of magnetic substances in finely divided form the particles of which are characterized by a very small and uniform grain size.

We have found that magnetic substances of the said kind may be obtained by preparing mixed crystals from oxides of magnetic metals and oxidic additional substances capable of forming mixed crystals with the said oxides, reducing the resulting mixed crystals and then mechanically comminuting the reduction product, as for example in an edge mill or ball mill. By the term mixed crystals are commonly meant solid solutions in which the crystal lattice of one component contains one or more other components of the solid solution. An oxidic additional substances there are chosen such as are more difficultly reducible than the employed oxides of magnetic metals. The oxides of the alkaline earth metals, including magnesium, or of aluminum or titanium, have proved especially suitable.

The presence of the additional substances in the form of mixed crystal components has the result that the magnetic substances, when being formed by the reduction of the mixed crystals, are produced and remain in a very finely crystallized state, because any rigid adhering together of the single small crystals and their recrystallization are prevented by the intercalation of the foreign substance.

According to our invention it is possible to produce for example finely divided powder of iron, nickel or the like and also powders of magnetic alloys, for example nickel-iron or nickel-iron-cobalt, when employing mixtures of oxides of several magnetic metals. Such a mixture is NiO—FeO—CoO and a suitable oxidic additional substance to form mixed crystals together with the mixture is in this case MgO. As another suitable mixture which also yields a magnetic nickel-iron-cobalt alloy may be mentioned $NiO.Fe_2O_3$—Fe_3O_4—Co_3O_4 and as additional substance Al_2O_3.

For the preparation of the mixed crystals it is preferable to start from a solution which contains a suitable compound both of the magnetic and the additional elements and to precipitate simultaneously the oxides of the magnetic metal and the oxidic additional substances. The obtained mixture is preferably subjected to a strong heat treatment in order to render the mixed crystal formation as complete as possible. In this manner a homogeneous mixed crystal is produced for example from the before-mentioned mixture NiO—FeO—CoO—MgO. By the following reduction which is preferably performed with hydrogen, a magnesium oxide containing nickel-iron-cobalt alloy is obtained.

Another method for the preparation of the mixed crystals consists in starting from the magnetic metals themselves and subjecting them after the addition of the foreign substances capable of forming mixed crystals, to an oxidizing fusion, the fused product then being reduced. By this method the above-mentioned aluminum oxide containing nickel-iron-cobalt alloy can be advantageously prepared. In most cases an addition of from 2 or 5 to 10 per cent of foreign substances is sufficient. Also greater amounts may be used, but they afford no particular advantages.

The metallic mass obtained by the reduction and comminution consists, even when the reduction has been effected at temperatures exceeding the recrystallization temperature of the magnetic metal, of very small crystallites the size of which may be down to 10^{-6} centimeter or less. For example by pulverizing iron prepared in the said manner there may be obtained an iron powder the particle size of which is $\frac{1}{1000}$ millimeter or less.

Magnetic substances prepared according to this invention are very suitable for the preparation of sound record carriers for electromagnetic sound recording and reproduction, for which purpose finely divided magnetic metals are embedded in a non-magnetic supporting mass or are applied to the same. They are also suitable for other electromagnetic purposes, as for example for the preparation of mass cores for Pupin coils, induction coils and the like.

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

Example 1

From a solution containing 10 per cent of cobaltous chloride and 0.5 per cent of magnesium chloride, after the addition of a small amount of hydrazine, the hydroxides of cobalt and magnesium are precipitated by means of caustic soda solution. The precipitate is washed and dried and heated for 2 hours at from 800° to 1000° C. and then cooled in an atmosphere of carbon dioxide. The mixed crystals of cobalt oxide and magnesium oxide thus formed are reduced at 400° C. with hydrogen. The reduction product is cooled in a stream of carbon dioxide to avoid spontaneous ignition and then ground

for a long time in a ball mill. The particles of the cobalt-powder-containing magnesium oxide thus obtained have a size of $\frac{1}{1000}$ millimeter.

Example 2

40 kilograms of pig iron with an addition of 3 kilograms of lime are burnt in a stream of oxygen. After cooling, the fused cake, consisting of mixed crystals of $CaO.Fe_2O_3$ with Fe_3O_4 in the spinel structure, is broken up into pieces of the size of nuts and reduced by means of hydrogen at a temperature of 600° C. An iron is thus obtained which contains CaO and a little unchanged $CaO.Fe_2O_3$ in fine, homogeneous distribution. The material is cooled in a stream of nitrogen and comminuted to a fine powder, first in an edge mill and then in a ball mill.

Example 3

A mixture consisting of 94 per cent of iron powder, 2 per cent of aluminum powder and 4 per cent of calcium oxide is burnt in a stream of oxygen. After cooling, the fused cake, is broken up and reduced with hydrogen at 500° C. The reduction product is cooled in a stream of hydrogen, then for a short time treated with a stream of carbon dioxide and finally comminuted to a fine powder as described in Example 2.

Example 4

A solution of 255 parts of potassium hydroxide in 3000 parts of water is added to a solution of 145.4 parts of $Ni(NO_3)_2.6H_2O$ and 404 parts of $Fe(NO_3)_3.9H_2O$ in 1000 parts of water. The mixture is boiled for 5 minutes. The precipitate thus produced is washed with water and after drying heated for 4 hours at 750° C. 20 parts of the obtained product which consists of $NiO.Fe_2O_3$, are mixed with 95 parts of iron powder and 2 parts of aluminum powder. The mixture is burnt in a stream of oxygen. The fused cake is then treated as described in Example 3. Thus an iron-nickel alloy with a nickel content of 5 per cent is obtained in the form of a very finely divided powder.

What we claim is:

1. The process of producing magnetic substances in finely divided form which comprises subjecting mixed crystals of at least one oxide of a magnetic metal and smaller amounts of at least one oxidic additional substance which is capable of forming mixed crystals with the said oxide and is more difficultly reducible than the said oxide, to such a reducing treatment as is sufficient for practically completely reducing the oxide of the magnetic metal, but insufficient for reducing the oxidic additional substance, and then mechanically comminuting the reduction product which contains the magnetic metal in reduced state.

2. The process of producing magnetic substances in finely divided form which comprises simultaneously precipitating from a solution at least one hydroxide of a magnetic metal and smaller amounts of at least one additional hydroxide which when in oxidic state is capable of forming mixed crystals with the oxide of the said magnetic metal, subjecting the precipitate to a heat treatment in order to produce the mixed crystals, reducing said oxide of a magnetic metal contained in the mixed crystals but not the additional oxidic substance and comminuting the reduction product.

3. The process of producing magnetic substances in finely divided form which comprises subjecting at least one magnetic metal together with smaller amounts of at least one additional substance which, when in the oxidic form, is capable of forming mixed crystals with the oxide of the said metal, to an oxidizing fusion, subjecting the fusion product to such a reducing treatment as is sufficient for practically completely reducing the oxide of the said magnetic metal contained in the fusion product, but insufficient for reducing the oxidic form of the said additional substance, and comminuting the reduction product.

4. As a new article of manufacture magnetic substances especially suitable for the manufacture of sound record carriers, in finely divided form which have been prepared in accordance with the process defined in claim 1.

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