Patented May 19, 1936

2,041,493

UNITED STATES PATENT OFFICE

2,041,493

PULVERULENT ALLOY

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No Drawing. Application January 19, 1934, Serial No. 707,339. In Germany January 24, 1933

17 Claims. (Cl. 75—122)

The present invention relates to a process of producing pulverulent alloys of metals capable of forming metal carbonyls.

Pulverulent alloys, as for example of iron with nickel, cobalt, chromium, molybdenum or tungsten, have hitherto usually been obtained by mechanical comminution of compact alloys. This method of working is lengthy and costly because as a rule such alloys are very tough, especially when they contain no carbon and oxygen. Moreover, the particles of powder readily become contaminated with undesirable foreign substances; their shape, also, is not uniform.

We have now found that pulverulent alloys of metals forming carbonyls 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.

The thermal decomposition of the carbonyl mixture, which is employed for example in the form of a mixture of two or more liquid or solid metal carbonyls, or a solution thereof, for example a solution of one or more solid metal carbonyls in one or more liquid metal carbonyls, and atomized or vaporized into the decomposition chamber, is preferably carried out at temperatures below 450° C. in order to avoid too strong a deposition of carbon. To produce the most uniform distribution possible of the carbonyl mixture, other gases, as for example carbon monoxide, carbon dioxide, nitrogen or ammonia, may also be introduced into the decomposition chamber, or the metal carbonyls may be atomized by means of such gases. In particular, decomposition in the hot free space of a heated vessel has proved advantageous because globular particles of uniform size are thus obtained throughout. The vessel may be heated either externally, or by introducing hot inert gases into the vessel, if desired in counter-current to the metal carbonyls introduced.

As coating "substances hindering fritting together" as previously mentioned, refractory substances, as for example oxides of alkaline earth metals, of magnesium, aluminium, beryllium and zinc and ceramic substances, silica, titanic acid and other difficultly fusible or difficultly reducible compounds, are used. It is not necessary to add these compounds as such to the metal powder; it is often preferable to use other compounds, for example nitrates which on heating yield the "substances hindering the fritting together". When working in a non-reducing atmosphere the "substances hindering the fritting together" may be prepared from the metals to be alloyed by slightly oxidizing the metal powder itself precedent to the heat treatment giving rise to the desired alloy formation.

The addition substances are preferably mixed as intimately as possible with the mixture of the pulverulent metals derived from carbonyls. A very intimate mixing is obtained for example by grinding the dry or wet mixture in a ball mill or by making into a paste and triturating the metal powder with a solution or suspension of the addition materials, as for example with a solution of magnesium or aluminium nitrate.

Small amounts of additional substances, as for example less than 1 per cent, are usually sufficient to prevent the agglomeration of the particles.

If it is desired to remove the additional substances from the metal powder after the heating, substances are chosen which are capable of subsequent removal by dissolution, as for example with dilute acids, by frothing or by magnetic separation in conjunction with wind-sifting.

Temperatures above 500° C. are suitable for the heating of the metal powders with the addition substances for the purpose of forming alloys; at temperatures below 500° C., the formation of alloys usually takes place incompletely and very slowly. It is preferable to work between 900° and 1000° C. The duration of heating may be shorter the higher the temperature employed, and the smaller the size of the particles of the metal powder. It is especially advantageous to carry out the heating of the metal powders in a current of hydrogen because in this way the diffusion within the particles of the metal powder is accelerated by the simultaneous volatilization of the carbon usually present.

The resulting alloy powders are distinguished by their purity and the globular shape of the single particles. The pulverulent alloys, especially powders of alloys of nickel-iron containing preferably between 40 and 80 per cent of nickel, or nickel-iron-cobalt, in particular such as contain between 25 and 50 per cent of iron, between 30 and 60 per cent of nickel and between 15 and 30 per cent of cobalt, or alloys of the latter kind containing up to 10 per cent of molybdenum or tungsten to increase the electric re-

sistance, have very good magnetic properties and consequently may be employed with special advantage for electromagnetic purposes, as for example for the preparation of mass cores for Pupin coils or in the radio industry. When preparing alloys intended for electromagnetic purposes, the hydrogen treatment for decarbonizing the powder is preferably carried on until the carbon content of the powder has fallen to 0.04 per cent or less.

Sometimes it may happen that the mass obtained in the manner described slightly coheres. In such cases merely a slight mechanical treatment, for example by shaking or if desired grinding for a short time will produce the pulverulent alloy aimed at.

The following example will further illustrate the nature of this invention but the invention is not restricted to this example. The parts are by weight.

Example

20 parts of a nickel-iron powder, consisting of 41.8 per cent of nickel, 1.4 per cent of carbon and the remainder iron, which has been obtained by the thermal decomposition of a mixture of nickel carbonyl and iron carbonyl in the hot free space of a heated vessel, are made into a paste with a solution of 1.2 parts of aluminium nitrate in 4 parts of water. The resulting mass is heated for 12 hours at 1000° C. in a stream of hydrogen. The aluminium nitrate is thus converted into aluminium oxide and the carbon content of the powder is lowered to 0.04 per cent. The frothy, easily friable mass obtained after the heating may be readily converted into an extremely finely divided powder with globular single particles by grinding in a ball mill. The ground product is heated again for about half an hour at 800° C. and then worked up into mass cores for Pupin coils in the usual manner.

What we claim is:—

1. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the metal particles of the resulting powder with a "substance hindering the fritting together" and then heating the coated powder in a non-oxidizing atmosphere at a temperature sufficiently high to effect alloying by diffusion within the particles of the metal powder without smelting.

2. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with up to 1 per cent by weight of a "substance hindering the fritting together" and then heating the coated powder in a non-oxidizing atmosphere at a temperature sufficiently high to effect alloying by diffusion within the particles of the metal powder without smelting.

3. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with a "substance hindering the fritting together" and then heating the coated powder in a non-oxidizing atmosphere at a temperature above 500° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

4. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with a "substance hindering the fritting together" and then heating the coated powder in a non-oxidizing atmosphere at a temperature between about 900° C. and about 1000° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

5. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with a "substance hindering the fritting together" and then heating the coated powder in a reducing atmosphere at a temperature above 500° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

6. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with a "substance hindering the fritting together" and then heating the coated powder in a hydrogen atmosphere at a temperature above 500° C. to effect alloying by diffusion within the particles of the metal powder without smelting and thus producing an alloy with a carbon content of at the most 0.04 per cent.

7. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with a refractory substance selected from the group consisting of the oxides of the alkaline earth metals, magnesium and aluminium and then heating the coated powder in a non-oxidizing atmosphere at a temperature sufficiently high to effect alloying by diffusion within the particles of the metal powder without smelting.

8. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, mixing the resulting powder with up to 1 per cent by weight of a refractory substance selected from the group consisting of the oxides of alkaline earth metals, magnesium and aluminium and then heating the resulting mixture in a hydrogen atmosphere at a temperature between about 900° and about 1000° C. to effect alloying by diffusion within the particles of the metal powder without smelting and thus producing an alloy with a carbon content of at the most 0.04 per cent.

9. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with at least one substance which on heating yields a "substance hindering the fritting together" and then heating the coated powder in a non-oxidizing atmosphere at a temperature sufficiently high to effect alloy-

ing by diffusion within the particles of the metal powder without smelting.

10. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with at least one substance which on heating yields a "substance hindering the fritting together" and then heating the coated powder in a non-oxidizing atmosphere at a temperature above 500° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

11. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with at least one substance which on heating yields a "substance hindering the fritting together" and then heating the coated powder in a reducing atmosphere at a temperature above 500° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

12. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, coating the resulting powder with at least one substance which on heating yields a "substance hindering the fritting together" and then heating the coated powder in a hydrogen atmosphere at a temperature between about 900° and about 1000° C. to effect alloying by diffusion within the particles of the metal powder without smelting and thus producing an alloy with a carbon content of at the most 0.04 per cent.

13. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, slightly oxidizing the particles of the resulting metal powder and then heating the powder in an inert atmosphere at a temperature sufficiently high to effect alloying by diffusion within the particles of the metal powder without smelting.

14. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, slightly oxidizing the particles of the resulting metal powder and then heating the powder in an inert atmosphere at a temperature above 500° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

15. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, slightly oxidizing the particles of the resulting metal powder and then heating the powder in an inert atmosphere at a temperature between about 900° and about 1000° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

16. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, slightly oxidizing the particles of the resulting metal powder and then heating the powder in a nitrogen atmosphere at a temperature sufficiently high to effect alloying by diffusion within the particles of the metal powder without smelting.

17. A process for producing pulverulent alloys of metals capable of forming metal carbonyls which comprises heating a mixture of at least two metal carbonyls at a temperature up to about 450° C. to decompose the carbonyls, slightly oxidizing the particles of the resulting metal powder and then heating the powder in a nitrogen atmosphere at a temperature between about 900° and 1000° C. to effect alloying by diffusion within the particles of the metal powder without smelting.

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