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METHOD OF PRODUCING METAL POWDER

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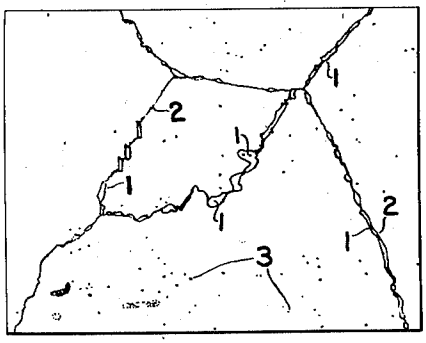


FIG. 1.

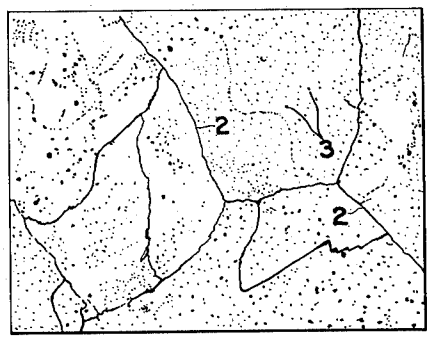


FIG. 2.

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METHOD OF PRODUCING METAL POWDER

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3 Claims. (Cl. 75—.5)

This invention relates broadly to the production of metal powder.

The desirable properties of alloys containing nickel, or nickel + cobalt, and chromium as the principal constituents, together with enough titanium and aluminum to form a precipitable phase with some of the nickel are well known. In particular, the alloys possess considerable resistance to heat, corrosion and creep, and so articles subjected in use to stress at high temperatures can advantageously be made of them. Now some articles can most conveniently be made by powder metallurgical methods but it is difficult or even impossible to reduce the alloys in question to powder by the methods used for some other metals and alloys. For instance they are too soft to be pulverised easily, they cannot be formed from the vapour phase and the need to prevent oxidation makes it difficult for them to be formed into powder by melting and then atomising them. Again, it is found that these alloys do not respond to a method by which stainless steel can be reduced to powder, namely heat treatment to cause precipitation of a carbide phase at grain boundaries and subsequent attack with, for example, a mixture of sulphuric acid and copper sulphate to dissolve the precipitated phase preferentially.

It is an object of the invention to reduce nickel-containing alloys of the kind set forth to powder by heat treatment followed by chemical attack.

The invention is based on the surprising discovery that a heat treatment followed by disintegration in a chemical solution, i. e. a process of the general kind known for stainless steel, can be used to reduce an alloy of the kind in question to powder if the heat-treatment is quite different from that applied to stainless steel.

The heat-treatment to which the alloy is subjected according to the invention comprises first heating the alloy to cause the precipitable phase to go into solution, cooling the alloy rapidly enough to maintain the solution substantially completely, and then so ageing the alloy as to harden it without causing carbide precipitation at the grain boundaries.

In the first stage of the heat-treatment the temperature should be 1100° C. or more, the upper limit being the incipient melting point of the alloy. It may be enough to maintain the alloy at this temperature for 5 minutes but a period of 15 to 30 minutes is preferred. Water-quenching is generally necessary in the cooling, but with very thin sections cooling in oil or even air may be enough. In the ageing step the temperature must not be high, since this encourages carbide precipitation at the grain boundaries, and should not exceed 800° C. A suitable temperature range is 650 to 750° C. with a duration of at least 2 hours, and the preferred treatment is heating for 16 hours at 700° C.

The different effects of applying to the alloys in question a heat-treatment of the kind applied to stainless steel and a heat-treatment according to the invention are illustrated by the accompanying drawings, in which the two figures are photomicrographs at a magnification of

750. Figure 1 shows an alloy of composition 19.2% chromium, 2.44% titanium, 1.09% aluminum and 0.04% carbon, with the remainder substantially all nickel, which has been heat-treated by heating for 1½ hours at 1200° C., slowly cooling in the furnace to 1050° C., air-cooling to room temperature and reheating for 16 hours at 700° C. As a result of this heat-treatment a carbide phase 1 has precipitated along the grain boundaries 2. In addition, within the grains there are precipitates 3 of a phase having the composition $Ni_3(TiAl)$, the ratio of titanium to aluminum in this phase being variable. The precipitated carbide phase renders the nickel-chromium alloy insensitive to chemical attack, i. e. it will not disintegrate in chemical solution. Thus when the nickel-chromium alloy in question has the microstructure shown in Figure 1, it is not reduced to powder by chemical attack, whereas it is only when stainless steel has a similar microstructure that it can be so reduced.

Figure 2 shows the same nickel-chromium alloy after heat-treatment according to the invention, i. e. heating for 20 minutes at 1150° C., water-quenching and reheating for 16 hours at 700° C. It will be seen that no carbide phase has precipitated along the grain boundaries 2, but there is some precipitation of the $Ni_3(TiAl)$ phase within the grains. The alloy with this microstructure is sensitive to chemical attack.

To disintegrate the alloy after the heat-treatment according to the invention, it is immersed in a mixture of hydrofluoric acid and an oxidising acid. This may be made by dissolving in 75 parts by volume of water 20 parts of 70% nitric acid (specific gravity 1.42) and 5 parts of 40% hydrofluoric acid. Although this acid mixture is strongly oxidising, no appreciable oxide film is formed on the surface of the powder particles, and these are immediately suitable for powder metallurgical use. The time of immersion in the solution depends upon the rate of disintegration, which will itself depend upon the temperature and strength of the solution. For example a sensitised sheet (20 s. w. g.) completely disintegrates to powder in 24 hours at room temperature and in 5 hours at 60° C. when immersed in fresh solution. With continued use the rate of disintegration becomes slower and longer times must be allowed, and eventually the solution becomes exhausted.

The invention can be applied to the alloy in any form, but strip, sheet or wire is preferred in order to present a large area for chemical attack. As is well known, the grain size of a wrought metal depends upon temperature and time of the solution heating, and hence by control of these factors some control can be effected over the size of the powder particles eventually produced. For a fine powder the lowest temperature and shortest time of heating should be used.

To avoid contamination of the powder, any oxide scale resulting from the heat-treatment operations should be removed before the alloy is subjected to the chemical attack.

Alloys to which the invention is applicable are those containing from 50 to 80% nickel + cobalt, the minimum nickel content being 40% and the maximum cobalt content being 30%, from 0 to 10% molybdenum, from 0 to 10% iron, from 0.1 to 8% titanium, from 0.1 to 8% aluminum and up to 0.25% carbon, with or without other elements commonly present in nickel-chromium heat-resisting alloys having a precipitable $Ni_3(TiAl)$ phase, the presence or absence of such other elements, e. g. manganese, silicon and zirconium, being immaterial in the present invention. In these alloys there must be enough titanium + aluminum to produce the precipitable phase, and to ensure success the minimum titanium + aluminum content should be 0.5%, though with less than this figure an alloy may respond to the process.

I claim:

1. A method of reducing to powder an alloy selected from the class containing nickel, or nickel and cobalt, and chromium as the principal constituents, the minimum nickel content being 40% and the maximum cobalt content being 30%, the total nickel and cobalt ranging from 50% to 80%, from 0 to 10% molybdenum, up to 0.25% carbon, from 0 to 10% iron, from 0.1 to 8% titanium, from 0.1% to 8% aluminum, and having a precipitable phase, said method comprising subjecting said alloy to heat treatment and disintegrating the heat treated alloy in chemical solution; said heat treatment comprising first heating the alloy to cause the precipitable phase to go into solution, cooling the alloy rapidly enough to maintain the solution substantially complete, and finally so aging the alloy as to harden it without causing carbide precipitation at the grain boundaries.

2. A method according to claim 1 in which the disintegration is effected by a mixture of hydrofluoric acid and an oxidising acid.

3. A method according to claim 1 in which said mixture is a solution of hydrofluoric acid and nitric acid.

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