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2,441,126

# UNITED STATES PATENT OFFICE

2,441,126

## OXIDATION RESISTANT ALLOYS

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3 Claims. (Cl. 75—22)

1

The present invention relates to alloys having a high resistance to oxidation and to the action of strong alkaline solvents such as fused alkaline nitrates. The alloy of the invention has particular advantages for use in making mounts for diamond dies.

Die mounts for hot drawing of wires must have a high resistance to oxidation. In drawing refractory metals, the drawing temperatures are frequently high enough to cause oxidation of the mounts at present in general use, to such a degree that the die will be loosened in the mount. Such a condition greatly impairs or even totally destroys the usefulness of the die, since the bearing in the die is no longer concentric in relation to the mount, nor is it any longer truly at right angles to the faces thereof. The axes of the bearings of the diamond nib and of the mount must coincide exactly. A high degree of accuracy in this respect is necessary and even a slight deviation from axial symmetry will greatly impair the usefulness and efficiency of the die. Again, in the drawing of tungsten or molybdenum, it frequently happens that a piece of wire breaks in the die and must be dissolved out. For this purpose, a fused alkaline nitrate is generally used. Such powerful reactants, however, also tend to cause oxidation and even to dissolve the mount metals generally employed and consequently to loosen the die nib in the mount with the deleterious effects hereinbefore described.

It is an object of the present invention to provide an alloy suitable for forming die mounts that will resist the oxidizing effects of temperatures encountered in hot drawing of refractory metal wires, such as tungsten or molybdenum wires and wires of other high melting point metals.

It is a further object to provide an alloy that will be suitable for use wherever it is necessary to prevent oxidation effects of high temperatures and of strong alkaline reactants.

Briefly described, the alloy of the invention consists of nickel, copper, chronium and silver combined in the proportions and according to the method hereinafter described. It must first be noted, however, that chromium, when heated either by fusion methods or by methods of powder metallurgy, has a marked tendency to oxidize and, in such case, not to alloy in a true sense with the other constituents noted. On the contrary, the chromium oxide forms nodes and these nodes are distributed through the mass of alloy of the other constituents in a porphyritic structure. Such a result is highly undesirable.

2

According to the method of the present invention, the chromium is first alloyed with nickel to form a nickel chromium alloy. This is effected by adding 90% nickel powder to 10% chromium powder, both of about 200 mesh or finer. The metal powders are thoroughly mixed as by ball milling until they are thoroughly commingled. The mixed nickel and chromium powders are then placed in a mold and compressed under hydraulic pressure of about 10 to 20 tons per square inch. The ingot so formed is then sintered in a hydrogen atmosphere at a temperature of about 300°–400° C. Great care must be taken to dry the hydrogen, as water vapor, which is present in nearly all commercially produced hydrogen, is a very active oxidizing agent and will cause the chromium to oxidize rapidly and to form the undesirable porphyritic structure above described instead of alloying with the nickel. The hydrogen may be dried by bubbling it through sulphuric acid or by passing it over phosphorus pentoxide.

The nickel and chromium may, however, be heat treated in any suitable non-oxidizing atmosphere other than hydrogen or the heating may be carried out in a vacum as in a high frequency vacuum furnace.

At the temperatures and under the non-oxidizing conditions mentioned, the nickel and chromium are sintered into a porous rod and are thoroughly diffused so as to form an alloy having a high resistance to oxidation. This rod is then broken down to powder form by mechanical means such as crushing or hammering. I call this alloy metal A.

A second batch of metal powders consisting of nickel powder 70% and copper powder 30% and also about 200 mesh or finer is thoroughly ball milled for several hours or until thorough distribution of the constituent powders is obtained. I call this metal, B metal.

A third batch of metal is then formed by mixing A and B metals in about the following proportions:

| | Percent |
|---|---|
| 20% metal A | 1 |
| 80% metal B | 99 |

I refer to this metal as metal C.

The final alloy is then formed by mixing from 75%–96% of powdered metal C with from 25%–4% of silver powder.

Assuming that the proportions of metal C consist of 10% of metal A and 90% of metal B, and assuming also that the final metal alloy is to consist of 90% of metal C and 10% silver, the

resulting very satisfactory alloy will consist of the following:

| | Percent |
|---|---|
| Nickel | 64.8 |
| Copper | 24.3 |
| Chromium | .9 |
| Silver | 10.0 |

The proportions given are by weight.

In my Patent 2,374,942 issued May 1, 1945, filed contemporaneously with this application, I have described the use of this metal for forming mounts for dies. The further treatment of the powdered metal above described, whether used for making die mounts or for other purposes, will be the same. The powdered metal is first poured into a mold of the desired shape and compressed in a hydraulic press under a pressure of 5–20 tons' pressure to the square inch.

The pressed compact is removed from the press and given a presintering treatment in a furnace in a dry hydrogen atmosphere at a temperature of from 300° to 400° C. for about one-half hour. A further and final sintering is then effected also in a dry hydrogen atmosphere at a temperature of from about 800°–900° C. for about a half hour. Under this treatment thorough diffusion and alloying of the constituents takes place without any trace of porphyritic characteristics and the metal shrinks to about 92% to 80% of the volume of the compacted mass.

Instead of chromium, zirconium, beryllium or aluminum may be substituted in the same proportion as the chromium without substantial change either in the method or in the characteristics of the final alloy.

Chromium, zirconium, beryllium and aluminum have the same properties in adding resistance to oxidation when combined with the other constituents of the alloy and should be alloyed separately with nickel in the same proportions as hereinabove stated for chromium alone to form the preliminary alloy. Thorough diffusion and alloying of nickel with these metals should be effected before adding to the other constituents if a true final alloy is to be obtained and a porphyritic structure is to be prevented. In any case, however, the proportions given above for the constituents are to be observed, viz: 75% to 96% of metal C powder with from 25% to 4% of silver powder. The proportions of the constituents of the final alloy will then be within the ranges of from 70.2% to 74% nickel, 0.1% to 2.0% of one or more of the group consisting of chromium, zirconium, beryllium or aluminum, and from 29.7% to 24% copper alloyed with silver in the proportions of 75% to 96% of the combined metals and 25% to 4% silver; or from 52.65% to 71.04% nickel, 0.075% to 1.92% chromium, zirconium, beryllium or aluminum, 22.275% to 23.04% copper and 25% to 4% silver.

Having thus described my invention, what I claim is:

1. Method of making an alloy that comprises first mixing a batch of finely divided metal powders consisting of 90% nickel and 10% chromium, compressing the same under hydraulic pressure and heat treating until thorough diffusion and alloying takes place, and finally grinding or crushing said alloy to a finely divided condition; then forming a second batch consisting of 70% nickel powder and 30% copper powder and thoroughly mixing the same; then combining and thoroughly mixing from 1%–20% of said nickel chromium alloy powder with from 99%–80% of said mixed nickel and copper powders; and then mixing from 75%–96% of said combined metal powders with from 25%–4% of silver powder; compacting the same under hydraulic pressure and heat treating the mass so compacted first at a temperature of 300°–400° C. and then raising said temperature to about 800°–900° C. and maintaining that temperature until all said constituent metals are thoroughly diffused, alloyed and sintered.

2. The method of making an alloy of nickel, chromium, copper and silver that comprises first separately forming an alloy of 90% nickel and 10% chromium, then combining from 1%–20% of said nickel chromium alloy in powder form with a batch consisting of 70% nickel powder and 30% copper powder, and then adding from 75%–96% of said combined metal powders to from 25%–4% of silver; thoroughly mixing said metal powders; compacting the same under hydraulic pressure and heat treating the mass so compacted until it is thoroughly sintered and until thorough diffusion and alloying of the constituent metals takes place.

3. The method of making an alloy of nickel, chromium, copper and silver that comprises combining from 1%–20% of a nickel chromium alloy powder consisting of 90% nickel and 10% chromium, with from 99%–80% of nickel and copper powders in the proportions of 70% nickel and 30% copper; then adding from 75%–96% of said combined powders to from 25%–4% of silver powders; compacting the same under hydraulic pressure, heating said compacted powders first at a temperature of about 300°–400° C. and then raising said temperature to about 800°–900° C. until the powders are sintered and the constituent metals are thoroughly diffused and alloyed.

JACOB KURTZ.


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## Certificate of Correction

Patent No. 2,441,126. May 11, 1948.

JACOB KURTZ

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Column 2, lines 45 to 47 inclusive for

| | Percent | | |
|---|---|---|---|
| 20% metal A------ | 1 | | 1%–20% metal A |
| 80% metal B------ | 99 | read | 99%–80% metal B |

and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 3rd day of August, A. D. 1948.

[SEAL]

THOMAS F. MURPHY,
*Assistant Commissioner of Patents.*

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