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METHOD FOR INTERNAL OXIDATION OF METAL POWDER FROM AN ALLOY, A METAL-POWDER MIXTURE OF VARIOUS ALLOYS OR A PARTIALLY ALLOYED METAL-POWDER MIXTURE

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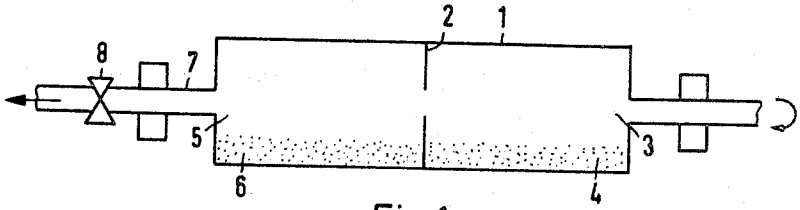


Fig. 1

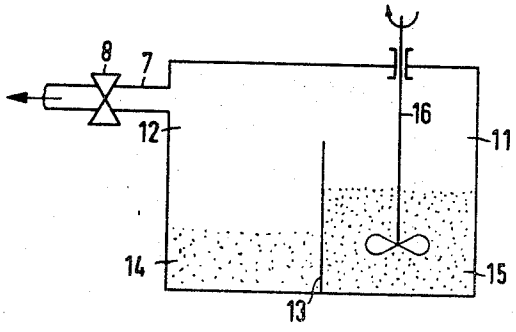


Fig. 2

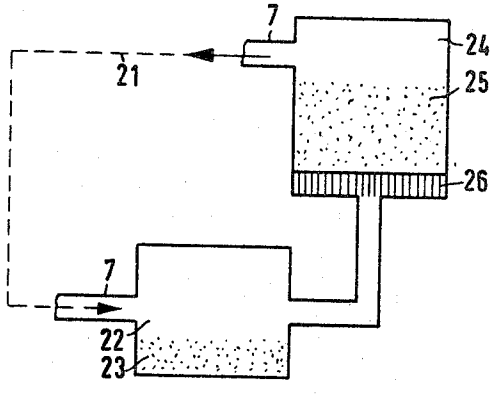


Fig. 3

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METHOD FOR INTERNAL OXIDATION OF METAL POWDER FROM AN ALLOY, A METAL-POWDER MIXTURE OF VARIOUS ALLOYS OR A PARTIALLY ALLOYED METAL-POWDER MIXTURE

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U.S. Cl. 75—.5 6 Claims

ABSTRACT OF THE DISCLOSURE

Described is a method of internal oxidation of metal powder from an alloy, a metal-powder mixture comprised of various alloys or a partially alloyed metal-powder mixture with a base metal which is less noble than silver. The method is characterized by internally oxidizing powder or powder mixtures located in one chamber of the two-chamber system, while heating a powder mixture comprised of a base metal and an oxide of the base metal, contained in the other chamber, to a partial dissociation of the base metal oxide.

Very finely dispersed oxide precipitations may be obtained in a metallic base by an internal oxidation of alloys of a relatively noble base metal as for example Cu, Ni, Fe and a less noble additional metal, such as Al, Mg, Si, Zr, Ti. The precipitated oxides increase the strength and the thermal stability, and stabilize the structure in addition to increasing the welding safety and the wearing quality.

The addition of non-noble metals must be selected with specific viewpoints in mind: The non-noble metal must be soluble in the base metal, in order to produce a fine oxide dispersion of which a certain minimum amount must be present in order to produce a sufficient incorporation of oxide. On the other hand, a specific limiting amount may not be exceeded in order to initiate the internal oxidation. The result of the internal oxidation furthermore depends upon the temperature and the oxygen partial pressure of the annealing atmosphere. The process is effected in the quickest manner when the partial pressure of the oxygen in the annealing atmosphere corresponds to the dissociation pressure of the oxide of the base metal, at the particular annealing temperature. A higher partial pressure leads to the formation of a braking cover layer of base metal oxide, a lower partial pressure also reduces the process.

The proper partial pressure may be established with reasonable certainty by embedding the sample pieces into a mixture comprised of the base metal and the base metal oxide, both preferably in powder form. This makes sense only for compact samples. On the other hand, the internal oxidation of compact samples of great thickness takes a very long time, as the speed of internal oxidation, depending on temperature and composition, is within the order of magnitude of several μ per hour. In order to obtain a finely dispersed oxide precipitation, the temperature should not be too high.

To produce larger workpieces from a completely internally oxidized material, it is preferred to use a powder metallurgy method:

A suitable alloy is pulverized mechanically, preferably by passage through a jet nozzle. In accordance with the present invention, the powder particles undergo an internal oxidation. These internally oxidized powders are then compressed by pressing and sintering, preferably by extruding.

Internal oxidation of powders of alloys having a base metal which is less noble than silver is even more difficult. According to a known method, such alloy powder is embedded into the powder mixture of a base metal and a base metal oxide. The subsequent separation of the internally oxidized alloying powder from the oxide powder which is either (a) not at all, (b) partially, or (c) completely reduced, is very difficult. Another known method is two-stage oxidation. First the powder is oxidized, for example in the presence of air, up to a defined weight increase, which results in an outer oxide layer. During a second annealing process, effected in a neutral atmosphere, the outside oxide layer is dissociated and the freed oxygen is diffused into the alloying powder. This method is not only complicated but is also difficult to control and regulate.

It is an object of the present invention to devise a method for the internal oxidation of metal powder from an alloy, from a metal-powder mixture out of various alloys, or from a partially alloyed metal-powder mixture having a base metal which is less noble than silver and has many advantages with respect to the afore-described method. According to the present invention, the powders or powder mixtures to undergo inside oxidation, which are contained in one chamber of a two-chamber system, and a powder mixture of a base metal and the oxide of the base metal which are located in the other chamber, are heated up sufficiently to cause a partial dissociation of the base metal oxide. Preferably, the temperature is so high that an oxygen partial pressure which is adequate for the internal oxidation and which corresponds to the dissociation pressure of the oxide, is obtained.

The method is effected in an arbitrarily designed, closed two-chamber system, for example in a sealed tube. During the heating process to required temperatures in an enclosed tube, for example in an electrically heated tubular furnace, a partial dissociation of the base metal oxide occurs and a partial oxygen pressure results which corresponds to the dissociation temperature of the oxide.

Heating of the pipe may be effected by means of an inside or an outside heating source. Electrical resistance heating is simplest to use, due to its easy technical execution.

In performing this method it is advantageous to supply both chambers with pulverulent material. The uniformity of internal oxidation may be promoted by moving the internally oxidizing alloying powder, during the annealing process. To this end, the pipe may be rotated or swung, whereby the powder is rolled around by baffle plates or deflectors.

Cooling is effected at the end of the annealing period required for a complete oxidation of the powder particles. The pipe may then be opened and the internally oxidizing powder removed. It is an advantage of the method of the present invention that with one and the same filling of base metal and oxide of the base metal, several loads of alloying powder may be internally oxidized.

In some cases it is preferable to produce a closed gas cycle. Inert gas may be introduced and the oxygen developing from the powder mixture of base metal and base metal oxide may be passed across the alloying powder.

Suitable alloying powders are, for example, Cu—Al, Cu—Ti, Ni—Al, Ni—Zr, Fe—Si. Suitable base metal oxide mixtures are, for example, Cu—Cu_2O, Ni—NiO, Fe—Fe_2O_3.

The use of a powder mixture comprising various alloying powders produces, following the processing of the internally oxidized mixture, a sinter working material, endowed with valuable characteristics. Also, using a partially alloyed powder mixture, i.e. a mixture whose heat balance of the alloying component, produces, following the powder-metallurgical processing of the internally

treatment did not yet lead to a complete concentration oxidized mixture, raw materials having a favorable structure.

The invention will now be further described with embodiment examples, illustrated by the figures, of which:

FIG. 1 shows one embodiment of our invention;

FIG. 2 shows an embodiment using an agitator; and

FIG. 3 shows a closed gas cycle embodiment.

In FIG. 1, a pipe **1** of heat-resistant material, for example heat-resistant steel, quartz, ceramic or refractory stone, is provided in the center or off-center with a separating wall **2**. Alloying powder **4** is inserted into the thus formed pipe portion **3**. The other pipe portion **5** contains a mixture **6** of base metal and oxide of the base metal, both in pulverulent form. The pipe **5** and **3** is evacuated through the tube **7** or rinsed with inert gas and subsequently sealed by closing valve **8**. Thereafter, the entire pipe is heated in an appropriate furnace to a temperature required for internal oxidation. The pressure balance is effected with a valve. During dissociation, the base metal oxide releases enough oxygen that the partial oxygen pressure is reached or maintained which remains at balance at the adjusted temperature. The oxygen which is needed for the internal oxidation of the less noble metal component of the alloying powder is supplied by the dissociating base metal oxide.

An example for the execution of the method of the present invention by using an agitator is shown in FIG. 2. Two chambers **11** and **12** are arbitrarily designed and separated from each other by a separating wall **13** and hold the powder mixture **14** comprised of base metal and oxide of the base metal and the alloying powder **15**. The latter is mixed by a mixer **16**.

FIG. 3 shows the performance of the method in a closed gas cycle. The gas current **21** passes through the decomposition chamber **22**, heated to the right temperature, which contains the mixture **23** of base metal and base metal oxide and is charged thereby with oxygen, corresponding to the partial oxygen pressure which is established at this temperature. The gas current or flow then enters the chamber **24** from below; this chamber contains the alloying powder **25** and creates a flowing bell **26** for the alloying powder.

The internally oxidized powders, obtained from the present method, may be used, for example, as initial powders for powder-metallurgically produced components and finished formed parts. However, larger cylinders may also be pressed and sintered therefrom, which are subsequently extruded into profiles. An additional application for the metal powder produced according to the method of the present invention is found in thickening by pressure or rolling, sintering and rolling for infinite tapes. The powder which is further processed by powder-metallurgical methods produces working materials having a uniform structure even across larger cross sections and very good qualities, especially high temperature qualities.

The following examples are given to further illustrate the present invention, without intent to limit the same.

EXAMPLE 1

A copper alloy with 0.8% Al by weight was pulverized by passage through a jet nozzle. Powder particles of a grain size of >0.5 mm. were filtered off. The remaining powder was placed into chamber **3** of FIG. 1. Chamber **5** contained a pulverulent mixture comprised by weight of 40% Cu_2O, 40% Cu and 20% sintered corundum. The entire system was heated to 875° C. and maintained at this temperature for 16 hours, with constant rotation of the pipe. By thermal dissociation of Cu_2O, a constant oxygen partial pressure of about $5 \cdot 10^{-3}$ torr occurred in the entire system. Thereby, the aluminum dissolved in the alloying powder oxidizes to Al_2O_3 which precipitates and forms a finely dispersed, thermally stable precipitation. The internally oxidized alloy powder was compressed into a cylinder and sintered at 800° C. This object was extruded into a rod. The thus obtained material combines good electrical conductivity of 50 m./Ωmm.² with high heat stability and is useful for welding electrodes.

EXAMPLE 2

A nickel alloy with 2% by weight beryllium was pulverized by passing through a jet nozzle, under a protective gas. The alloying powder was placed into chamber **24** in FIG. 3. Chamber **22** contained a pulverulent mixture comprised by weight of 40% NiO, 40% Ni and 20% sintered corundum. Both chambers were heated to 1000° C. and maintained at this temperature for 24 hours. The was charged with the constant oxygen partial pressure of about 10^{-7} torr. Highly purified argon, as a carrier gas, was charged with the constant oxygen partial pressure and created in chamber **24** a fluidized bed for the alloying powder. The beryllium, dissolved in the alloying powder, oxidized into BeO and formed a very finely dispersed precipitation The internally oxidized alloy powder was continuously compressed into a band, by means of step-by-step compression, and sintered at 900° C. By cold rolling, a dense, compact and good cold work hardenable material was obtained This material is useful for reactor components.

We claim:

1. A method of internal oxidation of metal powder selected from the group consisting of powdered alloys, a metal-powder mixture comprised of various alloys or a partially alloyed metal-powder mixture with a base metal which is less noble than silver, which comprises internally oxidizing the least noble constituent of said powdered alloy, metal powder mixture or partially alloyed metal-powder mixture in one chamber of a two-chamber system with molecular oxygen obtained from a simultaneous heating of a powdered mixture of a base metal and an oxide of said base metal, to a partial dissociation of the base metal oxide contained in the other chamber of said two-chamber system.

2. The method of claim **1**, wherein the heating is to such temperatures that a partial oxygen pressure, sufficing for internal oxidation, corresponding to the dissociation presure of the oxide, is obtained.

3. The method of claim **2**, wherein the heating is effected by an outside heating source.

4. The method of claim **3**, wherein both chambers are supplied with pulverulent materials.

5. The method of claim **4**, wherein the alloying powder is agitated.

6. The method of claim **4**, wherein the freed oxygen is passed through the alloying powder in a closed gas cycle.

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