PROCESS FOR THE PRODUCTION OF COPPER BODIES OF HIGH MECHANICAL STRENGTH AND HIGH ELECTRICAL CONDUCTIVITY

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PROCESS FOR THE PRODUCTION OF COPPER BODIES OF HIGH MECHANICAL STRENGTH AND HIGH ELECTRICAL CONDUCTIVITY

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ABSTRACT OF THE DISCLOSURE

Copper bodies with high mechanical strength at high temperatures are formed by producing copper powder under controlled conditions and compacting it under controlled conditions. The powder is produced as oxidised particles, with a mean diameter less than 10 microns, of copper of a purity at least 99.5% (not counting oxygen) and preferably containing at least 0.1% of carbon; the oxygen content of the powder is reduced to below 0.1% by heating in a reducing atmosphere without sintering the powder; and the reduced powder is then compacted under conditions that prevent substantial increase in oxygen content, the absolute maximum rise allowable being up to 0.3%.

This invention relates to the production of copper bodies by compacting copper powder and has as its object the preparation of such bodies possessing high mechanical strength at high temperatures, for example up to 600° C., together with good electrical conductivity.

The invention is based on the discovery that the fineness of the powder, the oxygen content of the powder, and the composition of the copper from which the powder is made all need to be controlled if this object is to be attained.

Copper powder suitable for use in the method of the present invention is in the form of finely divided particles of copper having an oxide coating, the copper being of a purity (not taking into account the oxygen content) of at least 99.5% and preferably containing at least 0.1% of carbon. Such powders will in general have an overall copper content (counting the oxygen as an ingredient) of at least 98.0%. By finely divided we mean having a mean particle diameter less than ten microns; we prefer to use copper powder having a mean particle diameter of about one micron.

In the method of the present invention the bodies are made from such powder by heating the powder in a reducing atmosphere to reduce the oxygen content of the powder to below 0.1% without sintering the powder and compacting the treated powder to form a billet or other body before any substantial reoxidation has occurred, that is to say before the oxygen content rises above 0.3%.

Copper powders for use in the method of the invention are commercially available, a preferred example being the grade of powder sold by Sherritt-Gordon Mines Ltd. of Fort Saskatchewan, Alberta, Canada under the name of NF—IM of 98.5% to 99% purity, counting oxygen as an impurity. It is thought that the carbon content of this powder is derived from an organic additive used to prevent agglomeration of the powder which becomes incor-

A suitable copper powder can also be made by precipitation from a solution of copper sulphate by the addition of sodium poly(acrylate). The liquor is decanted from the precipitate and the washed precipitate boiled with fresh copper sulphate solution, washed, then boiled in caustic soda to remove excess aluminium, washed and finally dried. The drying can be carried out in a reducing atmosphere to effect the necessary reduction.

Both powders have a carbon content between 0.1% and 0.2%, which is introduced into the copper during processing of the powder, the preferred upper limit of the carbon content being 0.2%.

The temperature to which the powders are heated to effect the required reduction in oxygen should be high enough to give a sufficiently short room temperature period to prevent sintering of the particulate material. Temperatures between 150° C. and 250° C. are satisfactory. A preferred temperature is about 200° C.

Suitable reducing atmospheres are:

Hydrogen
Mixtures of hydrogen and nitrogen, preferably one part by volume of hydrogen to three parts by volume of nitrogen or cracked ammonia
Town gas
Hydrocarbons, e.g., methane and propane.

Preferably the oxygen content is reduced to 0.05 to 0.07% by weight of the powder. The time taken to reduce the oxygen content to the required value will depend upon the initial oxygen content, upon the quantity and type of powder being treated and upon the type of furnace used and upon the reducing atmosphere employed and its flow rate through the furnace, and can readily be determined by experiment.

The treated powder is very prone to re-oxidise whilst in the deoxidised form but after being hot compacted to form a billet or other body it will not re-oxidise if stored at room temperatures. Accordingly unless the powder is to be compacted immediately after being de-oxidised it should be stored in a non-oxidising atmosphere.

The de-oxidised powder may be compacted to form a billet or other body by placing it in a press or mould and subjecting it to pressure, preferably to a pressure of about 5 tons/sq. in. (8 kg./mm.²) whilst the powder is at room temperature, maintaining the powder under such pressure whilst heating the press to a temperature of approximately 500° C. and then increasing the pressure to about 15 tons/sq. in. (24 kg./mm.²). Alternatively compaction of the powder may be effected by introducing it into the container of an extrusion press, compacting the powder in the container first at a pressure of about 5 tons/sq. in. (8 kg./mm.²) at room temperature and then, after heating the container to about 500° C., applying a pressure sufficient to extrude the compacted billet to form a rod or other elongated body. The hot pressed billet or extruded rod may be rolled and/or drawn into wire or strip.

At the end of the hot press or extrusion stage the product of the invention is very substantially harder than tough pitch copper either as cast or when fully cold worked, the relative H.V.10 values being:

Cast tough pitch copper (as cast) 50
Cast tough pitch copper (when fully cold worked) 115
Product of the invention 150
The high Vickers Hardness of the product of the invention is substantially retained when the product is heated up to 500° C. The ultimate tensile strength of wire or rod produced from copper powder containing carbon by the process according to the invention exceeds 40 tons/sq. in. (65.0 kg./mm.²) as compared with tough pitch copper whose ultimate tensile strength is about 30 tons/sq. in. (47.0 kg./mm.²). After heating the former up to 600° C. for one hour, its ultimate tensile strength still exceeds 30 tons/sq. in. (47.0 kg./mm.²) whereas that of cold worked tough pitch copper is reduced to 15 tons/sq. in. (24 kg./mm.²) by heating for one hour to a temperature of between 150° C. and 250° C. The electrical conductivity of the product of the invention is 90% to 95% of that of the International Annealed Copper Standard (I.A.C.S.) and this degree of conductivity is maintained after heating for one hour at 600° C.

The product of the invention may be used with advantage for many purposes where high mechanical strength and high conductivity are required to be maintained under high temperature conditions. Examples are high temperature cable conductors, resistance welding electrodes, soldering bits, commutator bars, small switch parts and the like some of which articles can be formed directly by hot pressing the de-oxidised copper powder in appropriately shaped dies.

The accompanying drawing is a graph comparing the effect of heat on the tensile strength of copper wires made by the method of the invention with a copper wire made from electro-refined tough pitch copper.

The vertical readings show the ultimate tensile strength at room temperature, both in tons per square inch (right hand figures) and kg. per sq. mm. (left hand figures) and the horizontal readings represent the temperature in degrees centigrade at which the wire was preheated before testing.

Details of the wires tested are given in the table below. The rapid falling off in tensile of the wires of Examples II and III at high temperatures is attributed to the rather high oxygen content. Wire made from copper powder not containing carbon or from copper powder mechanically mixed with graphite showed less improvement in tensile strength under the specified conditions over tough pitch copper. It is preferable so to control the oxygen content that it does not rise above 0.1% during forming, since excess oxygen makes fabrication difficult owing to a reduction in ductility of the powder, this entails initially reducing the oxygen content below this value and then rapidly consolidating the powder before substantial oxidation takes place.

<table>
<thead>
<tr>
<th>Curve</th>
<th>Control</th>
<th>Example I</th>
<th>Example II</th>
<th>Example III</th>
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<tbody>
<tr>
<td>Wire made from</td>
<td>Electro-</td>
<td>Barenth</td>
<td>Powder precipitated in sodium poly-</td>
<td>Powder precipitated in sodium poly-</td>
</tr>
<tr>
<td></td>
<td>refined</td>
<td>Sherritt</td>
<td>acrylic at 65° C</td>
<td>acrylic at 65° C</td>
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<tr>
<td></td>
<td>tough</td>
<td>Garden</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>pitch</td>
<td>powder.</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>copper.</td>
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Although it is believed that the residual oxygen layer on the surface of the powder particle contributes to the high mechanical strength of the copper bodies after heating it is believed that the carbon content of the copper from which the particles are made is also an important factor.

The wire of the control and the wires of Examples I, II and III were all made by conventional processes by rolling and drawing from a billet, the billets used in Examples I, II and III being made from the specified powders by the process described above entailing an initial compacting at room temperature followed by the application of a higher pressure at 500° C.

What I claim as my invention is:

1. A method of producing copper bodies consisting of compacted copper powder which comprises the steps of: heating in a reducing atmosphere at a temperature of 150° C.-250° C. a powder in the form of finely divided particles of copper having a mean particle diameter less than 10 microns and of 99.5% purity containing at least 0.1% of carbon and having an oxide coating, for a time sufficient to reduce the oxygen content of the powder to below 0.1%; and then compacting the powder while it carries a thin oxide coating but before any substantial re-oxidation occurs.

2. A method as claimed in claim 1 in which the powder is compacted before the oxygen content rises above 0.1%.

3. A method as claimed in claim 1 in which the oxygen content is initially reduced to 0.05% to 0.07%.

4. A method as claimed in claim 1 in which the powder is compacted before any substantial reoxidation occurs by applying compacting pressure to the powder at room temperature and raising the temperature to sintering temperature while the pressure is maintained.

5. A method as claimed in claim 4 in which the powder is compacted at room temperature at a pressure of about 5 tons/sq. in. (8 kg./mm.²), the temperature of the powder is raised to 500° C., and the pressure is then raised to about 15 tons/sq. in. (24 kg./mm.²).

6. A method as claimed in claim 4 in which the powder is compacted by filling the chamber of an extrusion press with the powder, applying pressure to the powder at room temperature in the chamber of the press by operating the press below extrusion pressure and then raising the temperature of the powder and the pressure to extrude the compacted powder from the press as an elongated body.

7. A method of producing copper bodies consisting of compacted copper powder which comprises the steps of: processing the powder in such a way as to introduce carbon into the copper and to produce in the powder in the form of finely divided particles of copper having a mean particle diameter less than 10 microns and of 99.5% purity containing at least 0.1% of carbon and having an oxide coating; heating said powder in a reducing atmosphere to a temperature between 150° C. and 250° C. for a time sufficient to reduce the oxygen content of the powder to below 0.1%; and then compacting the powder while it carries a thin oxide coating but before any substantial reoxidation occurs.

8. A method as claimed in claim 7 in which the powder is compacted before the oxygen content rises above 0.1%.

9. A method as claimed in claim 7 in which the oxygen content is initially reduced to 0.05% to 0.07%.

10. A method as claimed in claim 7 in which the powder is compacted before any substantial reoxidation occurs by applying compacting pressure to the powder at room temperature and raising the temperature to sintering temperature while the pressure is maintained.

11. A method as claimed in claim 10 in which the powder is compacted at room temperature at a pressure of about 5 tons/sq. in. (8 kg./mm.²), the temperature of the powder is raised to 500° C., and the pressure is then raised to about 15 tons/sq. in. (24 kg./mm.²).

12. A method as claimed in claim 10 in which the powder is compacted by filling the chamber of an extrusion press with the powder, applying pressure to the
powder at room temperature in the chamber of the press by operating the press below extrusion pressure and then raising the temperature of the powder and the pressure to extrude the compacted powder from the press as an elongated body.

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A. J. STEINER, Assistant Examiner.