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PROCESS FOR IMPROVING THE MECHANICAL PROPERTIES OF COPPER USING A REFRACTORY DISPERSED FILLER

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This is a division of application Serial No. 657,506, filed May 7, 1957, and now abandoned.

This invention relates to processes for increasing the tensile strength, fatigue strength, and resistance to high-temperature creep of copper metal and to the copper of improved properties so produced. The invention is more particularly directed to processes in which a hydrous, oxygen-containing compound of copper is deposited upon a water-insoluble refractory filler insoluble in copper and selected from the group consisting of metal oxides having a melting point above 1100° C. and a free energy of formation at 1000° C. of from 30 to 150 kcal. per mole per oxygen atom in the oxide, the filler being in the form of particles having an average size less than 1000 millimicrons and the volume loading of filler being about from 0.05 to 20% calculated on the amount of copper in the deposited oxygen-containing copper compound, thereafter the copper compound is reduced to copper while maintaining the temperature in the range of from 200 to 500° C., and the reduced product is compacted to a dense mass of metal having the refractory filler uniformly dispersed therein and having an apparent density which is from 90 to 100% of the absolute density.

Copper metal products containing uniformly dispersed refractory particles, which can be produced according to methods of the present invention, are claimed in our copending United States patent application Serial No. 220,894 filed August 31, 1962, now Patent No. 3,143,789.

Copper is a preferred metal for many uses because of its ductility and conductivity. In some uses, however, its softness and its property of losing tensile strength as the temperature is raised to 800 or 900° F. are serious detriments. Hitherto it has not been known how to harden copper and improve its strength, particularly at elevated temperatures, and still retain its desirable ductility and conductivity. It has been especially noted that attempts to effect improvement in these properties have been accompanied by a marked decrease in the electrical conductivity of the copper. Moreover, improvements in strength heretofore achieved have been rapidly lost at elevated temperatures.

It is therefore an object of this invention to provide copper having improved tensile strength and resistance to creep and fatigue, particularly at elevated temperatures. Another object is to provide such improvements in the properties of copper without substantial loss in its conductivity or ductility. Another object is to provide processes for producing copper having such improved properties. Further objects will appear hereinafter.

Now according to the present invention it has been found that the foregoing objects are accomplished by processes in which a hydrous, oxygen-containing copper compound, such as copper oxide or copper hydroxide, is deposited upon particles of a refractory filler insoluble in the copper, the copper compound is reduced to copper metal at a temperature in the range of 200 to 500° C. and the product so obtained is compacted to a dense mass having the refractory filler uniformly dispersed therein, the degree of compaction being sufficient to give the metal

an apparent density which is from 90 to 100% of the absolute density. The filler must be in the form of particles having an average size less than 1000 millimicrons and must consist of a metal oxide having a melting point above 1100° C. and a free energy of formation at 1000° C. of from 30 to 150, preferably 75 to 150, and still more preferably 110 to 150 kilogram calories per mole. The volume loading of filler is about from 0.05 to 20% calculated on the amount of copper in the deposited oxygen-containing copper compound.

By the processes of the invention metallic copper is produced having exceptional hardness and tensile strength without substantial loss of ductility or electrical conductivity. The properties of the product indicate that the filler is uniformly dispersed through the metal. By uniformly dispersed is meant that the filler particles are present inside the grains of the copper metal as well as at the grain boundaries. This is in contrast to the products made by so-called "internal oxidation" of alloys of copper with silicon or aluminum, where the filler particles are concentrated at grain boundaries resulting in a brittle product having poor fatigue properties. Thus the advantage of having the filler uniformly dispersed is that the strengthening effect of the particles is obtained without sacrificing ductility or resistance to fatigue. The fact that the filler particles are uniformly dispersed in the products of this invention can be shown by an electron micrograph using replica techniques, by which it can be seen that the particles are present inside the grain as well as at the grain boundaries.

As the filler one may employ metal oxides having the characteristics hereinafter described. Typical materials of this class include silica, alumina, zirconia, titania, magnesia, the rare earth oxides including thoria, and zinc oxide. With care, iron oxide, cobalt oxide or nickel oxide can also be used. Mixed metal oxides, such as metal silicates can be used, zircon being a specific example. Suitable silicates include magnesium silicate, aluminum silicate, calcium silicate, iron silicate and nickel silicate.

Especially preferred oxides are those having the highest free energy of formation. Broadly, the oxide must have a free energy of formation of from 30 to 150 kcal. per mole per oxygen atom in the oxide, but the oxides which have free energies of 75 to 150 or, still better, from 110 to 150 are particularly preferred. Thus, magnesia, alumina, thoria, and other rare earth oxides are among the most preferred materials to use as fillers according to this invention.

The oxide particles must be in the size range below one micron. As with the metalloids, it is preferred that the particles be in the size range from 5 to 250 millimicrons an especially preferred range being from 10 to 250 millimicrons. The particles should be dense, discrete, and anhydrous for best results. Particles which are substantially spheroidal or cubical in shape are also preferred, although anisotropic particles such as fibrous platelets can be used used for specific effects. Anisotropic particles produce metal compositions of lower ductility, however, and in those instances where ductility is desired, particles approaching isotropic form are preferred.

Colloidal metal oxide aquasols are particularly useful as a source of the fillers in the desired finely divided form. For example, silica aquasols such as those described in Bechtold and Snyder U.S. Patent 2,577,485 are especially useful as a source of silica particles for use in the processes of the invention. Zirconia sols are similarly useful. The art is familiar with titania sols, these being described, for instance, by Weiser in "Inorganic Colloid Chemistry," vol. II, "Hydrous Oxides and Hydroxides," for example, can be used to advantage. Sols of alumina, thoria, magnesia, and rare earth oxides are especially preferred for

use where the copper is to be subjected to sustained high temperatures in service.

Suitable particulate metal oxides can also be produced by thermal processes such as by burning metal chlorides. For instance, by burning aluminum trichloride, titanum tetrachloride, or zirconium tetrachloride to produce the corresponding oxide, under conditions adapted to give the product as discrete individual particles or aggregated structures which can be dispersed to such particles, products are obtained which are useful in processes of the invention.

Having selected a suitable filler as above-described, one then deposits upon the dispersed filler particles a hydrous, oxygen-containing compound of copper. The copper compound can be, for instance, copper oxide, copper hydroxide, hydrous oxide of copper, copper oxylate, oxycarbonate, or hydroxycarbonate. The volume of deposited copper compound is large relative to the volume of filler particles. An especially suitable method of depositing the copper compound is to precipitate it from a solution of a soluble copper salt such as copper nitrate, copper chloride or copper sulfate.

The above-mentioned precipitation can be accomplished conveniently by adding the soluble salt to an aqueous, alkaline solution containing the filler particles, while maintaining the pH above 5. An especially suitable method is to add simultaneously but separately a soluble metal salt, a colloidal aquasol containing the filler particles, and an alkali such as sodium hydroxide, to a heel of water. Alternatively, the sol of filler particles can be used as the heel, and the metal solution and alkali added simultaneously but separately thereto.

As the alkali, hydroxides, such as sodium hyroxide, potassium hydroxide or ammonia can be used. Similarly, carbonates such as ammonium carbonate or sodium carbonate can be employed. Because of their volatility, ammonium compounds are preferred.

It is preferred not to coagulate or gel the sol of filler particles during the deposition process. By simultaneously adding the filler and the copper salt solution to a heel as already described, this danger is minimized. By employing dilute solutions the chances of coagulation and precipitation are also diminished.

It is desirable that during the deposition process the filler partciles be essentially surrounded with the oxygen-containing copper compound, so that when reduction occurs later in the process, aggregation and coalescence of the filler particles is avoided. In other words, it is preferred that the ultimate particles of the filler be not in contact with each other in the product from the deposition step. It will be understood that in addition to the precautions already mentioned vigorous mixing and agitation are helpful in insuring dispersion of the filler particles.

After the hydrous oxygen compound of copper is deposited upon the filler, any salts formed during the deposition are removed by washing. If ammonia was the alkali used in the deposition and the ammonium salts thus formed are volatile, salt removal may be accomplished simply by heating without washing. The precipitated copper compound containing the filler particles is ultimately dried at a temperature of about 100° C., but it is possible to dry the product, remove soluble salts, and again dry the product if necessary.

Ordinarily in processes of this invention, relatively large amounts of hydrous compounds of copper will be applied as a coating to a relatively small amount of filler. The amount of coating material will vary somewhat with the particle size of the filler and especially with the surface area of the filler. In general from 0.05 to 20 volume loading percent of the filler in the final metal composition is desired. When the filler particles are very small, for instance when they have a surface area greater than $20/D$ square meters per gram, D being the density of the filter in grams per milliliter, volume loadings of from .5 to 5% are preferred.

The precipitated copper-oxygen compound on the filler particles is next reduced to copper metal by any suitable reduction technique. The art is familiar with methods whereby copper oxides can be reduced and any such method can be employed. Conveniently, the copper-oxygen compound can be reduced by subjecting it to contact with a stream of hydrogen at elevated temperatures, although mixtures of hydrogen and nitrogen, carbon monoxide, hydrocarbon gases and other gaseous reducing agents can also be used.

The temperature of reduction suitably can be in the range from 200 to 500° C., preferably 200 to 400° C. With low volume loadings, temperatures above 400° C. cause excessive sintering of the product and hence when sintering is not desired, high temperatures are avoided.

After most of the reduction has been effected, however, higher temperatures can be tolerated in the process. Thus the product can be placed in the furnace at a controlled temperature and hydrogen gas slowly added. The temperature is gradually raised during reduction so that after most of the oxygen has been removed, the temperature is raised into the range from 600 to 900° C. However, when working with such high temperatures excessive sintering is always a danger and it is desirable to avoid such sintering because it leads to densification difficulties later on in the process. Especially when ferrous metal oxides are used as fillers, very mild reducing conditions should be used.

After reduction is complete the powder preferably is pressed into a dense billet. Prior to pressing the copper should be protected to avoid reoxidation.

The pressing of the reduced metal powder into a billet is preliminary to the final compaction. The compaction is effected under such conditions that the final apparent density is from 90 to 100% of the absolute density, preferably from 98 to 100%. The compaction is carried out by subjecting the reduced powder to very high pressures, preferably at a temperature of about 700 to 800° C. In some instances, it is desirable to heat to even higher temperatures, such as 1000° C., during the compaction step. To obtain optimum properties it is also preferred to hot- or cold-work the resulting product, as, for example, by rolling, hot-extruding or similar techniques well-known in the metallurgical art. The rolling or compacting technique is continued until the density of the product is in the range desired.

The products of this invention comprise dispersions, in copper metal, of particles of a water-insoluble refractory filler, insoluble in the copper and selected from the group consisting of metal oxides having a melting point above 1100° C. and a free energy of formation at 1000° C. of from 30 to 150 kcal. per mole per oxygen atom in the oxide. The filler is non-reducible—that is, it is an oxide which is not appreciably reduced by copper at a temperature of 1000° C.

The filler in the compositions of the invention must be in the size range below 1000 millimicrons, preferably from 5 to 250 millimicrons, and still more preferably from 10 to 100 millimicrons. Although very small filler particles can be employed in the processes of this invention, they are difficult to handle because they sinter easily when the final products are dried, and they gel easily in the liquid phase. Thus, while 5 millimicron particles can be used and will be found to be present in the products of the invention, the problems involved are greatly simplified when larger particles—say 10 millimicrons—are used. When the filler particles reach about 25 millimicrons in size, they are considerably more difficult to coagulate or gel, and also more difficult to sinter during the heating associated with the reduction and compaction. Particles having an average size of 100 millimicrons are still within the colloidal range and hence can be handled without problems involving settling, particularly during the process of making the compositions of this invention. Thus, products containing filler particles

in the size range from 25 to 100 millimicrons can be easily and readily produced directly from colloidal aquasols and hence such products are preferred compositions of the invention.

Products in which the filler particles are substantially discrete, dense particles of a refractory oxide also are preferred compositions of the invention. Alumina, magnesia, thoria, hafnia, and other rare earth oxides are especially effective fillers in products which are subjected to very high temperatures during preparation or use. On the other hand, zirconia, titania, silica, and other oxides give excellent results when used in processes employing the lower ranges of temperatures heretofore described for making products of the invention.

Preferred also are products in which the filler is in the form of essentially isotropic particles—that is particles which approach spheroidal or cubical shape. It is particularly desirable that the filler particles be anhydrous in the novel compositions.

Volume loadings of the filler in the novel copper compositions are in the range of from 0.05 to 20%. Below 0.05 there is little effect, and about 20% the mass is extremely hard to handle because of loss of ductility. Moreover, with more than 20% volume loading it is difficult to keep the filler particles from coming into contact and sintering. Volume loadings from 0.1 to 10% are preferred with 2.5 to 5% being especially preferred.

The particles of the filler in the products are substantially completely surrounded by a coating of copper which maintains them separate and discrete. The particles are isolated and do not come in contact with each other. Thus coalescence and sintering of the filler material is prevented. In other words, the compositions comprise a continuous phase of metallic copper containing dispersed therein the non-reducible oxide filler. That the copper metal is essentially the continuous phase can be demonstrated by the electrical conductivity of the mass, since this conductivity is essentially unaffected by the presence of the oxide, when calculated on a volume percent of copper.

A technique for showing that the filler particles are uniformly dispersed in the copper metal matrix involves examining a sample of the copper metal for tensile strength, yield strength, hardness and ductility. Thus, for example, in the novel products the tensile strength measured in any direction is equivalent to that measured in any other direction. In contrast, dispersions heretofore prepared by powder metallurgy show laminations or striations of the particles scattered in the direction of working as, for instance, in the direction of cold rolling when this method of compaction is used. In such instances the strength and ductility in the transverse direction may be high, but if measured in a perpendicular direction to the transverse direction lower values will be found. Products of the present invention do not show these laminations on hot or cold working, nor do they show variations in tensile strength, ductility, hardness or ultimate yield strength as a function of direction of measurement of these properties. The nature of the filler phase in the copper metal can also be shown, in those cases where the filler is insoluble in nitric acid, by dissolving away the metal and measuring the insoluble particles so obtained by conventional techniques. The copper metal dissolves easily in nitric acid and the copper nitrate and excess nitric acid can be removed by dialysis, for instance, from such filler particles as silica. The resulting dispersion of silica in the remaining aqueous medium can be characterized by electromicrographs or by determining particle size by sedimentation techniques using Stokes' law.

This uniformity of dispersion also characterizes the filler particles in the copper powder after reduction but before compaction. Such products hence are a novel aspect of the present invention. These compositions can be used as a masterbatch in making powder metallurgical products. For example, copper powder containing 10 volume percent zirconia can be diluted with other copper powder and formed, by powder metallurgy into various shapes, including wires, which are useful in electrical applications.

The products of this invention are not stable above the melting point of the metal phase. For this reason, they should not be melted or heated to a temperature at which the continuous copper phase becomes molten, since melting will cause the finely divided oxide to become aggregated.

As previously noted, in the products of the invention, the filler particles are uniformly dispersed in the copper metal. As a result, ductility and resistance to fatigue can be maintained at relatively high volume loadings of filler. Thus products containing 2.5 volume percent of filler have very good ductility and even at 10 volume percent ductility is acceptable, in most cases. Compositions containing more than 10 volume percent filler are useful mainly as a masterbatch for dilution with other powdered metals as above-described.

The invention will be better understood by reference to the following illustrative examples.

Example 1

In this example, "Ludox" Colloidal Silica, a commercially available silica sol, was used as the source of the filler material. The reactor consisted of a stainless steel cylinder with a conical bottom, fitted with connections whereby fluid could be pumped from the bottom of the reactor through a ½" pipeline and back to the reactor. Feed solutions could be introduced into the system through three separate T-tubes in the external line.

The feed solutions consisted of (a) 5 liters of copper nitrate containing 15 moles of copper, (b) 5 liters of aqueous ammonia containing 22.5 moles of NH_3, and (c) 3.6 grams of "Ludox" Colloidal Silica (30% SiO_2, SiO_2:Na_2O 90, silica particles 17 mμ, spherical and discrete) diluted to 5 liters. These feed solutions were introduced into the reactor (containing 25 liters of water), simultaneously but separately, at a rate of 275 ml./min. for each solution. The pH of the final slurry was 5.8.

The precipitate of $Cu(NO_3)_2.3Cu(OH)_2$ on the silica particles was recovered by filtration and washing. The product was dried at 250° C., whereupon a CuO-SiO_2 product was formed. This product was reduced at 400° C. in a slow stream of hydrogen in a period of 5 hours, over 95% of the oxygen of the CuO being removed. The reduction was continued for three hours at 600° C. The final product was compacted in a press at 20 tons/sq. inch into a 1" billet which was finally forged and cold rolled to 1/16" thickness. At this stage, the mass had reached 99% of theoretical density—that is, the apparent density was 99% of the absolute density.

Example 2

The reactor used to prepare the precipitate of copper hydrous oxide on the colloidal oxide filler consisted of a stainless steel tank with a conical bottom. The bottom of the tank was attached to stainless steel piping, to which were attached three inlet pipes through T's. This circulating line was connected to a centrifugal pump of 20 g.p.m. capacity. From the pump the line returned to the tank.

Initially, the tank was charged with 5 gals. of water. Equal volumes of three solutions containing the desired quantities of reagents were then added into the middle of the flowing stream through ⅛" diameter tubing attached to the T tubes. These solutions were added at uniform equivalent rates over a period of about one-half hour. Through the first T was added a solution of copper nitrate; through the second, aqueous ammonia; through the third, the colloidal oxide (zirconia) filler. Two gallons of each reagent was used, including a 3-molar copper nitrate solution, a 4.6 N ammonia solution, and a zirconia aquasol stabilized with nitrate, containing 14 g.

of ZrO_2. The zirconia sol contained anhydrous, colloidal particles in the size range from 10 to 100 millimicrons, with the average particle diameter being about 65 millimicrons.

The solutions were added into the reactor simultaneously while the pump was in operation. The rate of addition was controlled uniformly by flow meters. The pH of the solution in the tank was taken at frequent time intervals to insure proper operating. The final pH was 6.5. The slurry was circulated for a few minutes after the addition of the reagents had been completed, and then the solution was pumped into a filter. Approximately 95% of the copper was recovered as hydroxide precipitate. The precipitate was filtered, washed with water, and dried at a temperature of about 200° C. for 72 hours.

The product was then placed in an oven at a temperature of about 100° C., and a mixture of argon and hydrogen was slowly passed over the dried powder. This gas stream had been carefully freed of oxygen, and had been dried. The temperature in the furnace was slowly raised over a period of an hour. The flow of hydrogen was then gradually increased and the temperature in the furnace was raised until a temperature of 500° C. was reached, whereupon a large excess of hydrogen was passed over the sample in order to complete the reduction. Finally, the temperature was raised to 600° C. while continuing to pass hydrogen over the sample. In this way, a finely divided copper powder containing 2.5 volume percent zirconia was produced.

The copper powder was cold-pressed to 80% of theoretical density, using a pressure of 40 tons per square inch. It was then sintered at 900° C. in vacuum to 91% of theoretical density and finally hot rolled at 800° C. to a reduction in thickness of ½. After annealing at 400° C. the Rockwell E hardness was 87, the ultimate tensile strength was 45,000 p.s.i. and the elongation was 25%, whereas a copper metal control containing no filler dropped in hardness from about 98 to less than 40.

Metallurgical examination of the ZrO_2-filled copper revealed a fine grain structure, and pictures at 750 magnification gave no evidence of the filler, showing that the zirconia was present in the copper as a uniform dispersion in a very finely divided state.

Example 3

A zirconia sol containing particles 10 millimicron average size was used in place of "Ludox"; otherwise the process of Example 1 was followed.

A billet having a loading of 2.5 volume percent was prepared. After reduction, it was noted that the control copper containing no filler had sintered considerably, whereas the loaded sample had not.

Samples of this powder were compacted at 25 tons per square inch pressure. The resulting bars were sintered at 900° C., and then hot rolled at about 700–800° C. to a reduction in thickness of ½. The hardness of this material was 93 R_E, and remained at this value after annealing at 800° C.

On cold rolling, the hardness rose to 102 R_E, but dropped to 81 after annealing at 800° C.

This material can be hot-extruded into copper wire, and in this form is an excellent conductor of electricity for use at temperatures of 400° C. or even above.

Example 4

A copper product was made in a manner identical to that in Example 3, except at 10 volume loading.

Hardness values of 101 were obtained on hot working, and the value did not change after heating at 800° C. On cold rolling, the hardness rose to 103, and dropped back to 101 after annealing at 800° C.

Example 5

Example 4 was repeated, using a colloidal aquasol of titania containing 25-mμ particles,

Samples obtained on hot rolling had a hardness of 96, and 94 after annealing at 800° C. On cold rolling, hardness rose to 98, but fell to 95 after annealing.

Example 6

This sample was similar to Example 1, except that 100-mμ silica particles (range 50 to 200 mμ), in the form of an aquasol was used. Twenty grams of silica was thereby incorporated into 900 grams of copper.

The resulting copper powder was hot pressed and finally hot extruded at 590° C. from a 1" die through a ¼" opening. This copper had a Rockwell E hardness of 89 and had theoretical density. The hardness did not change after annealing at 800° C.

Another sample of the copper powder was hot pressed at 950° C. and 3000 p.s.i. in order to yield a billet 2" x ¼" x ¼", having 97% of theoretical density. This material was likewise extremely hard.

Example 7

Using the technique of Example 1, a dispersion of Al_2O_3 in copper was prepared, using an aquasol of colloidal Al_2O_3 in place of the "Ludox." The reduced material was compacted at 25 tons per square inch, sintered at 900° C. for 2 hours, and finally hot rolled to 50% of original thickness. In the product the alumina was present as 30 millimicron particles, uniformly dispersed throughout the copper, at a volume loading of 0.5 percent. The product had a Rockwell E hardness of 83, a value which did not change on annealing at 800° C. On cold rolling, the hardness rose to 99 and then dropped to 66 on annealing at 800° C.

Example 8

Using the technique of Example 7, a dispersion of ThO_2 particles in copper was prepared. This dispersion contained 1 volume percent of 15 millimicron thoria particles. On long term aging at 900° C., this product retained its structure better than products described in the previous examples. Cold rolling or annealing at 800° C. did not change the hardness of the hot-rolled copper.

We claim:

1. In a process for producing copper having improved high-temperature strength the steps comprising depositing a hydrous, oxygen-containing copper compound upon a water-insoluble refractory filler, the filler being in the form of particles, insoluble in copper, having an average size below 1000 millimicrons anda melting point above 1000° C. and being a metal oxide having a free energy of formation at 1000° C. of from 30 to 150 kcal. per mole per oxygen atom in the oxide, and the volume loading of filler being about from 0.05% to 20% calculated on the amount of copper in the deposited oxide, and thereafter reducing the deposited coating to metallic copper while maintaining the temperature in the range from 200 to 500° C. throughout the entire mass, and compacting the reduced product to a dense mass of metal having the filler dispersed therein and having an apparent density which is from 90 to 100% of the absolute density.

2. In a process for producing copper having improved high-temperature strength the steps comprising depositing a hydrous, oxygen-containing copper compound upon a water-insoluble refractory filler, the filler being in the form of particles, insoluble in copper, having an average size in the range of 5 to 250 millimicrons and a melting point above 1000° C. and being a metal oxide having a free energy of formation at 100° C. of from 30 to 150 kcal. per mole per oxygen atom in the oxide, and the volume loading of filler being about from 0.1% to 10% calculated on the amount of metal in the deposited oxide, and thereafter reducing the deposited coating to metallic copper, while maintaining a temperature of less than 1000° C. throughout the entire mass, and compacting the reduced product to a dense mass of metal having the

oxide filler dispersed therein and having an apparent density which is from 90 to 100% of the absolute density.

3. A process of claim 2 in which the filer is a metal oxide and initially is in the form of particles having an average size in the range of 12 to 100 millimicrons colloidally dispersed as an aquasol, the oxide being selected from the group consisting of alumina, thoria, magnesia and zirconia.

4. A process of claim 1 in which the deposited oxygen-containing copper compound is partially reduced with hydrogen to metallic copper at a temperature in the range of 200 to 500° C., and thereafter reduction is completed at a temperature of 600 to 900° C.

5. A process of claim 1 in which reduction of the deposited oxygen-containing copper compound to metallic copper is continued until the total oxygen content of the composition is in the range from 1.0 to 2.0 times the oxygen content of the original oxide filler.

6. A process of claim 1 in which the compacting of the reduced product is continued until the metal becomes a continuous phase.

7. In a process for producing copper having improved high-temperature strength the steps comprising (1) oxidizing a metal chloride of the group consisting of aluminum trichloride, titanium tetrachloride, and zirconium tetrachloride to produce the corresponding oxide as filler particles having an average size below 1000 millimicrons, (2) depositing upon said filler particles a hydrous, oxygen-containing copper compound, the volume loading of said filler particles being about from 0.5% to 20% calculated on the amount of copper in the deposited oxygen-containing copper compound, (3) thereafter reducing the deposited coating to metallic copper while maintaining the temperature in the range from 200 to 500° C. throughout the entire mass, and (4) compacting the reduced product to a dense mass of metal having the filler dispersed therein and having an apparent density which is from 90 to 100% of the absolute density.

8. A process of claim 7 wherein compaction step (4) is carried out at a temperature in the range of 700 to 800° C.

9. A process of claim 7 wherein the metal chloride oxidized in step (1) is aluminum trichloride, whereby the filler oxide is aluminum oxide.

10. In a process for producing copper having improved high-temperature strength the steps comprising (1) oxidizing a metal chloride of the group consisting of aluminum trichloride, titanium tetrachloride, and zirconium tetrachloride to produce the corresponding oxide as filler particles having an average size below 1000 millimicrons, (2) depositing upon said filler particles, a hydrous, oxygen-containing copper compound, the volume loading of said filler particles being about from 0.5% to 20% calculated on the amount of copper in the deposited oxygen-containing copper compound, (3) thereafter reducing the deposited coating to metallic copper while maintaining the temperature in the range from 200 to 500° C. throughout the entire mass, (4) compacting the reduced product, at a temperature in the range of 700 to 800° C., to a dense mass of metal having the filler dispersed therein and having an apparent density which is from 90 to 100% of the absolute density, and (5) mechanically working the compacted metal mass.

11. A process of claim 10 wherein the metal chloride oxidized in step (1) is aluminum trichloride and the mechanical work of step (5) consists of hot rolling at a temperature below 800° C., followed by cold rolling at a temperature below 800° C.

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