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FEATHERY COPPER POWDER AND PROCESS OF PRODUCING THE SAME

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This invention relates to copper powder having new and improved properties and to the production of such powder.

In the electro-plating art as practiced heretofore the object has usually been to deposit metal from an electrolyte on a cathode in the form of a plate, such plate often being referred to as reguline metal. In such processes the metal is deposited without any material or extensive liberation of gases at the cathode. Frequently conditions in the plating bath change during the process and sludge or mushy metal is accidentally produced which has been recognized as a condition highly undesirable in the production of reguline metal and the sludge or mushy metal has been discarded as useless and the conditions corrected as quickly as possible to produce a reguline metal again.

It has been proposed heretofore to vary the conditions in the plating bath to deposit metal on the cathode in finely divided form as contrasted with the usual reguline form for certain purposes, for example, where the finely divided material is later to be flattened or stamped out into sheet-like particles for making metallic paint. The prior art metal powders are characterized by being composed of a mixture of particles of various and radically different sizes and shapes, a large proportion of these particles being in the form of slender rods having rounded nodules thereon or dense, compact bodies. These prior powders when compressed under several thousand pounds pressure per square inch produce an article of relatively low tensile strength, elongation and hardness.

So far as I am aware, however, it has never been proposed prior to my present invention to carry out an electro-plating process under such conditions that but very little if any of either of the foregoing products will be produced but that metal will be deposited in finely divided feathery form which can subsequently be compacted or compressed into an article having relatively high hardness, tensile strength and other physical properties. The copper powder produced by my present invention is of an entirely new character or form and possesses new and much improved properties. This powder is composed of particles substantially all being of the same shape and size and with an almost entire absence of small particles of other shapes and compact bodies. This powder is soft and feathery or flocculent, that is, each particle has a body of irregular or fixed or characteristic shape with rough edges and sharp pointed extremities and while these shapes vary somewhat they are all generally similar. For example, when copper particles of this invention are viewed in profile under the microscope they are jagged and may be said to resemble the edges of a Killarney fern. These feathery particles possess the property of matting together under pressure and may be compressed or worked into various shapes and articles by known processes involving compression, heat treatment and the like but without requiring any binder and when so compressed or shaped the resulting article is dense, relatively non-porous and has a much higher tensile strength, elongation and hardness than similar articles made of the prior powder and containing the slender rod particles or the dense compact bodies.

This property of matting together is traceable largely, I believe, to the fact that my particles are in a flocculent condition which permits a close interlocking and interweaving of the particles as contrasted with the lack of such action where the rounded compact particles of the prior art are used. This contrast may be visualized by thinking of compressing felt on the one hand and sand on the other hand.

My new powder is distinctive and readily recognized but since it is not so easily described in brief language I have employed the terms "Koehler metal powder" or "feathery powder" to describe the powdered metal of my invention and wish it to be understood that powder of my invention is referred to herein whenever either of these expressions are employed.

I have discovered that when a suitable electrolyte is electrolized under certain conditions and with the evolution of gas over practically all of the active cathode surface that Koehler metal powder will be deposited

on the cathode without any reguline metal or any appreciable amount of the prior art powder being deposited. The conditions which must be observed in attaining my results involve several factors including (1) the amount of metal in solution in the electrolyte (2) the acidity of the bath (3) the voltage and amperage of the current (4) the temperature of the bath (5) the distance between electrodes and (6) the nature and amount of any additional metal or metals present in solution in the electrolyte.

While these various factors may vary widely it is important that the conditions be such in all cases to cause the evolution of gases over the active face of the cathode.

Various copper salts may be used in producing feathery metal according to my present invention, for example, sulphates or chlorides or acetates of copper. Obviously many other salts may be employed but in all instances I prefer that the solution should be an acidulated solution.

The amount of the metal which should be in solution in the electrolyte for the production of the feathery powder may vary between about ½ percent by weight and upward to an amount at which under the same current conditions the evolution of gas over the cathode practically ceases. This maximum is usually only a few percent. If the solution contains more than the maximum amount of the metal salt the excess salt will polarize the anode and seriously interfere with the economical operation of the process. The weight of powder per unit volume increases as the metal in solution increases.

The acidity of the bath may vary from about ½ of 1 percent by weight and upward therefrom but for the higher efficiencies the acid content should not exceed about 10 percent. In general the increase in the acid content above the minimum is accompanied by a drop in voltage and amperage efficiency without any attendant and compensating results.

The electrolyzing current employed may vary widely above certain readily determined minimum limits. However, practical commercial considerations which involve power and equipment cost as well as the character of the deposit will determine the upper limits of the voltage and amperage. It will be understood that the current density required will increase as the amount of metal in solution in the electrolyte increases and the voltage will decrease with an increase in the acidity of the electrolyte until about 10 percent of acid is reached after which it will remain substantially constant. The voltage will also vary with the distance between electrodes, that is, the voltage will be lower the closer the electrodes are together and higher the farther apart they are placed. The voltage and ampere efficiency is perhaps highest when the acidity of the electrolyte is below about 6 percent. In general the amperage should not fall below about 30 or exceed about 240 per square foot of cathode area.

Variations in current also result in variations in the weight per unit volume of the powder.

The temperature of the bath may vary upwardly from about 35 degrees F. to about 200 degrees F. and naturally tends to increase as the electrolysis proceeds unless steps are taken to cool the electrolyte. As the temperature increases the mobility of the ions increases and hence the current density must be increased to prevent the formation of reguline metal or nodular powdered metal. Practical considerations make it advisable to maintain the bath temperature below about 150 degrees F. and above about 70 degrees F. and exceptionally good results can be obtained between about 70 degrees F. and about 100 degrees F. The size of the particles varies with the temperature and the weight per unit volume increases as the temperature rises.

The distance to be maintained between the electrodes will largely be controlled by practical considerations such as current cost and the space available for and the cost of the equipment. A distance of about 3″ between electrodes gives good results but this distance may be increased or decreased if desired and is preferably decreased.

While I may practice my invention in a simple acidulated electrolyte containing in solution only the copper to be converted into feathery form, I find it advantageous and in some respects desirable also to employ in the solution a metal which is more positive in nature according to the electro-motive scale than the copper. Many different metals may be employed. Merely as illustrative of such metals I mention sodium and zinc. These more electro-positive metals should be used in the form of salts of the same acid as is present with the copper.

These added salts may vary in amount through a considerable range which is limited beginning with about ½ of 1 percent by weight and extending upwardly thereabove to the extent of a few percent.

I cite the following as a specific example of the conditions under which my new and improved copper powder may be produced. An electrolyte is prepared containing in solution between about ½ and about 3½ percent by weight of copper, preferably about 2 percent, and between about ½ and about 10 percent by weight of free acid, preferably about 6 percent. The temperature of the bath may range between about 35 degrees F. and about 200 degrees F. but preferably is kept between about 70 degrees F. and about 100 degrees F. The electrodes are spaced apart about 3″. The current may range between about 40 amperes and 240 amperes per

square foot of cathode area with about 70 amperes being preferably with the above preferred conditions. The voltage may vary from 7 to 2 depending on the acid content with about 2 volts being suitable under the above preferred conditions.

When operating under the above preferred conditions, Koehler metal powder will be deposited on the cathode. This powder should be completely removed periodically from the cathode for otherwise the deposit changes rapidly and contains an increasingly large content of dense compact copper of the prior art. This change is apparently controlled by the current density and length of deposition period between the steps of removal of deposit from the cathode. In general these variables may be summarized by the statement that a current flow of about 12 ampere hours per square foot of cathode area between the steps of removal gives satisfactory Koehler metal powder; that is employing about 70 amperes for a ten minute interval. However, it will be understood that this is a preferred combination of the foregoing conditions and that they may vary widely therefrom, for example, from about 1 to about 70 ampere hours.

The complete removal of the deposit from the cathode as just mentioned is preferably accomplished by a severe operation, such as brushing, which dislodges any particles which tend to adhere closely to the cathode. Jolting or jarring operations are not satisfactory for they do not remove all the deposit and hence permit the formation of the dense compact prior art powder.

As an illustration of the use of an added, more highly positive metal in the electrolyte in producing Koehler copper powder the electrolyte may contain in solution about 2 percent by weight of copper, and about 2 percent by weight of sodium or zinc and about 6 percent by weight of free acid. With this illustration as in the preceding example, sulphuric acid is used and the metals are in the form of sulphates. With the anodes and cathodes spaced apart about 3″ and the temperature of the bath maintained between the foregoing ranges Koehler copper powder will be produced when a current of 70 amperes per square foot of cathode area is used and when the voltage is maintained at about 3 volts.

For the successful practice of my invention the composition of the bath must remain substantially constant particularly with reference to the concentration of the metal to be converted into feathery powder for, if on the one hand, the concentration of the copper increases and approaches the saturation point of the liquid the anode will become polarized by crystallization thereon of the salt of that metal and will thus require that the voltage be largely increased with a consequent power loss, and, on the other hand, if the concentration of acid in the bath increases materially the predominant effect of the electrolysis will be the liberation of hydrogen and consequently a reduction in the yield of feathery metal.

Under the normal conditions of operation of my process which includes the continuous evolution of gases over substantially the entire active surface of the cathode, the cathode efficiency as measured by the amount of metal deposited is materially lower than the anode efficiency as measured by the amount of acid radical liberated. This difference in efficiency may and often does amount to as much as 20 percent. Consequently in order to maintain a balance between the amount of copper salt and the amount of acid in the bath some means must be provided to compensate for this difference in efficiencies.

One means for maintaining the said balance is to employ a combination of anodes, a part of which are inactive so far as their reaction with the free acid radical in the bath is concerned and others of which are active with such acid radicals. Such anodes are not claimed in this application, however, but this subject matter is being reserved for presentation in a separate application.

Koehler metal powder or the feathery powder as described herein feels soft and smooth to the touch and the particles are highly oriented and highly voluminous in bulk.

They are homogeneous, highly compressible and exceptionally pure chemically and have an enormous expanse of filiform surface which provides for the intimate mixture of the particles with each other or with other substances which gives the material properties which collectively make the feathery powder susceptible to treatment of many and varied kinds and utilization to many useful purposes.

This application is a continuation in part of my copending application, Serial No. 182,866 filed April 11, 1927, and 190,341, filed May 10, 1927, being restricted to copper powder and its production. No claim is made herein to the remaining subject-matter of these three applications.

Having thus described my invention so that those skilled in the art may be enabled to practice the same what I desire to secure by Letters Patent is defined in what is claimed, it being understood that the foregoing detailed disclosure has been made by way of illustration only and not by way of limitation on the scope of the invention as defined in what is claimed.

I claim:

1. The process of producing the herein described feathery copper powder which comprises electrolyzing an acidulated solution containg above about ½ per cent by weight of copper, under such conditions as will

evolve gas substantially continuously over the entire active surface of the cathode thereby depositing the copper in a feathery powdered state on the cathode and removing the deposit from the cathode at a rate that uniformity is substantially maintained in the feathery form of the deposit.

2. The process as described in claim 1 in which the solution contains between about ½ percent and about 6 percent by weight of copper.

3. The process as described in claim 1 in which the solution contains a few percent by weight of a metal of a more highly positive nature than copper.

4. The process as described in claim 1 in which between about ½ percent and about 10 percent by weight of free acid is present.

5. The process as described in claim 1 in which the solution contains between about ½ percent and about 10 percent by weight of acid and between about ½ percent and about 6 percent by weight of a metal of a higher positive nature than copper.

6. The process as described in claim 1 in which copper is present in the solution in an amount between about ½ percent and about 6 percent by weight and in which solution is also present between about ½ percent and about 6 percent by weight of a higher positive metal and also between about ½ percent and about 6 percent by weight of free acid.

7. The process of producing the herein described soft feathry powder of copper, which comprises electrolyzing an acidulated solution containing above about ½ percent by weight of copper, with a cathode current density too high to deposit reguline metal, and that will produce a substantially continuous evolution of gas over the entire active surface of the cathode, thereby depositing the copper as a soft feathery powder on the cathode, and removing all the deposit from the cathode before the total current flow has reached an amount sufficient to produce hard non-feathery particles in the deposit.

8. The process as described in claim 7, in which the removal of the deposit from the cathode is accomplished periodically with sufficient frequency to prevent the formation of non-feathery particles after a current flow of between about 10 and about 20 ampere hours per square foot of cathode area.

9. As a new article of manufacture, copper powder of the herein described feathery form having the properties and characteristics of feathery powder produced by electrolyzing an acidulated solution containing above about ½ percent by weight of copper, under such conditions including current voltage and density as evolve gas substantially continuously over substantially the entire active surface of the cathode and removing substantially all deposit from the cathode at such a rate that uniformity is substantially maintained in the feathery form of the deposits.

10. As a new article of manufacture, copper powder of the herein described feathery form having the properties and characteristics of feathery copper powder produced by electrolyzing a solution containing between about ½ percent and about 6 percent by weight of copper under such conditions including current voltage and density as will evolve gas substantially continuously over the cathode and removing substantially all deposit from the cathode at such a rate that uniformity is substantially maintained in the feathery form of the deposit.

11. As a new article of manufacture, copper powder of the herein described feathery form having the properties and characteristics of feathery copper powder produced by electrolyzing a solution containing between about ½ percent and about 6 percent by weight of copper, between about ½ percent and about 6 percent by weight of sodium and between about ½ percent and 10 percent by weight of acid under such conditions including current voltage and density as will evolve gas substantially continuously over substantially the entire active surface of the cathode, and removing the material deposited on the cathode at such a rate that uniformity is substantially maintained in the feathery form of the deposit.

12. As a new article of manufacture, copper powder having the properties of that produced by the process of claim 7.

13. As a new article of manufacture, copper powder having the properties of that produced by electrolyzing an acidulated solution containing about ½ percent by weight of copper, with a cathode current density too high to deposit reguline metal, and that will produce a substantially continuous evolution of the gas over the entire active surface of the cathode, thereby depositing copper as a soft feathery powder on the cathode, and removing all the deposit from the cathode after a current flow of between about 10 and about 20 ampere hours per square foot of cathode area, thereby preventing the formation of non-feathery particles.

In testimony whereof I hereunto affix my signature this 4th day of January, 1930.

WILLIAM KOEHLER.