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APPARATUS FOR PRODUCING METALLIC POWDERS

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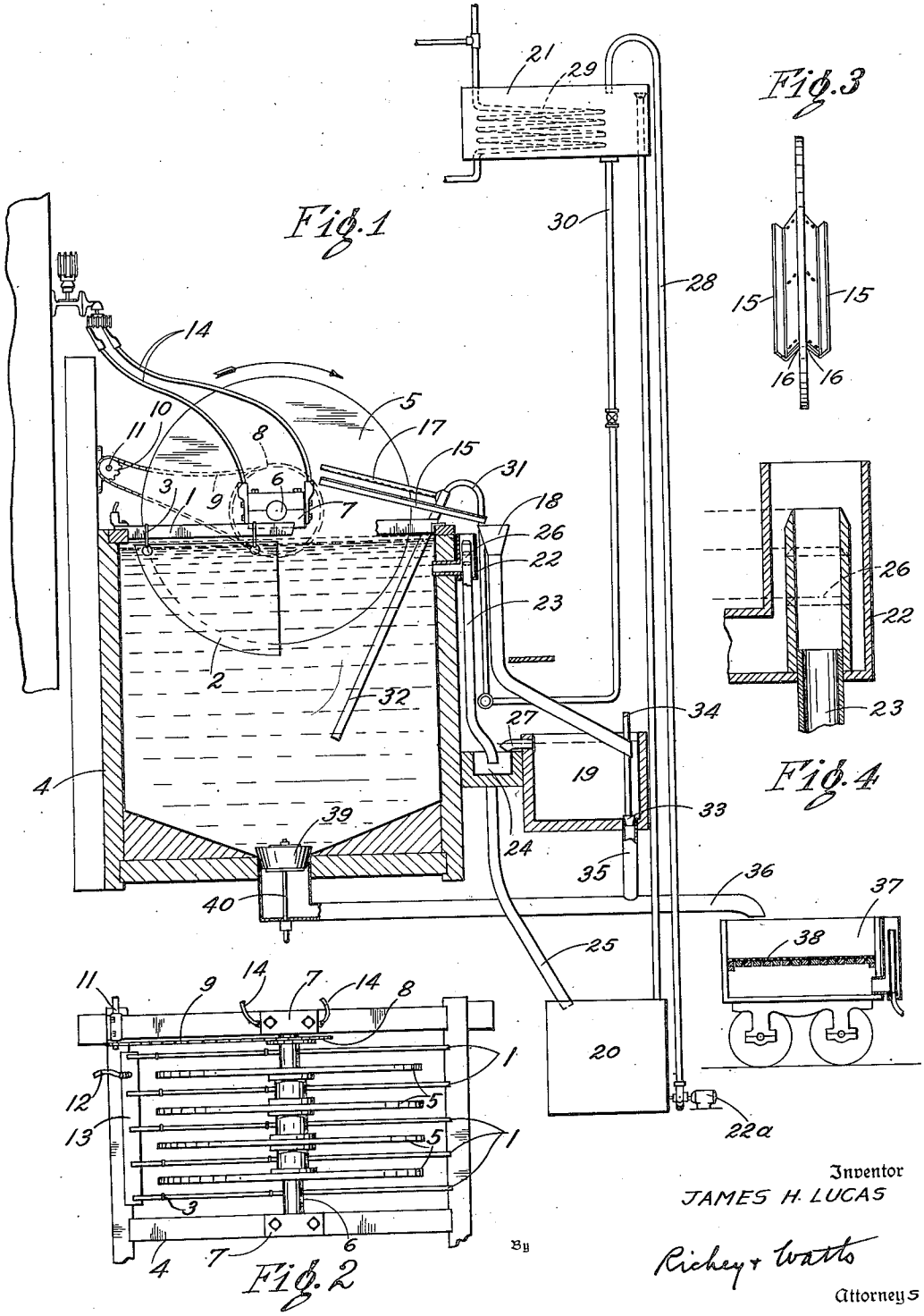


Fig. 1

Fig. 2

Fig. 3

Fig. 4

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APPARATUS FOR PRODUCING METALLIC POWDERS

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Original application September 26, 1930, Serial No. 484,540. Divided and this application March 20, 1934, Serial No. 716,492

8 Claims. (Cl. 204—5)

This invention relates to apparatus for producing metal powders.

A method and apparatus used heretofore for this purpose included spaced cathode rods and anodes dipping into an electrolyte bath adjacent said rods. The bath was an electrolyte of copper sulphate and sulphuric acid. Current passing through the electrolyte from the anodes to the cathode rods deposited copper on these rods in a fluffy mass or slime. The deposit on the rods was allowed to build up for about 15 minutes and at 15 minute intervals the rods were given a jarring by hand which caused at least some of the deposit to fall off and into the bottom of the electrolyte bath from which it was ultimately removed, and after washing and drying was screened to secure a copper powder of the desired particle size.

This old system presented disadvantages which may be briefly summarized as follows:—The building up of the copper deposit on the rods caused the rods to increase in size, thus changing the density of the current at the cathode. The density of current determines the particle size of the copper deposit on the rods and if the density changes as is the case where the deposit on the rods is constantly changing, the size of these particles varies. When the particles, therefore, were jarred off of the rods, particles of varied size were recovered from the bottom of the electrolyte bath. The variation in density of the current caused some large crystals to form along with those of a smaller size, thus necessitating sieving to remove the large crystals. According to the old system where it was desired to obtain crystals which would pass through a 200 mesh or better, with the jarring of the rods every 15 minutes it was found that about 20% of the total copper powder produced consisted of crystals too large to pass through the 200 mesh sieve. In addition it was necessary to use substantially pure copper anodes due to the fact that any impurity separated from the anodes during the course of deposition would fall into the bottom of the electrolyte bath and become mixed with the copper powder jarred from the rods where a substantially pure copper powder was desired. It was, therefore, necessary to start off with anodes of relatively high purity in order to avoid the presence of these impurities in the resulting copper powder. In addition in the old system the rods required replacing about once a week due to the fact that at the point where the rods entered the electrolyte, oxidation by the old system occurred with eating away of the rods at that point until they were finally eaten through and dropped to the bottom of the tank.

An object of the present invention accordingly is to do away with these difficulties and to provide a simple and efficient apparatus for producing metal powders such as copper powder which shall be simple, relatively inexpensive to operate, requiring little supervision, doing away with the necessity for renewal of parts as in the old system. Other objects will be in part obvious and in part pointed out hereinafter.

The invention accordingly comprises the novel apparatus and combinations thereof, specific embodiments of which are described herein by way of example only and in accordance with the manner in which I now prefer to practice the invention.

This application is a division of my copending application Serial No. 484,540, filed September 26, 1930, now Patent No. 1,959,376.

In the accompanying drawing wherein I have shown a preferred embodiment of my apparatus—

Figure 1 shows a somewhat diagrammatic side elevation, partly in section, of an apparatus embodying my invention;

Fig. 2 is a plan view corresponding to Fig. 1 and showing the relative positions of anodes and cathodes;

Fig. 3 is a detailed view of the spray used for washing away the copper powder scraped off the cathodes; and

Fig. 4 is a somewhat enlarged detailed view of a device for controlling the overflow of electrolyte from the electrolyte bath.

Referring now to the drawing, I shall describe the apparatus as applied to the production of copper powder of a desired degree of purity and containing a copper content which may be as high as 99.75%. It is to be understood, however, that the invention herein is not restricted to apparatus for producing copper powder but is applicable generally to production of other metallic powders which are susceptible to electro-deposition in powder form, such as iron, silver, tin, lead and zinc.

The numeral 1 designates a series of spaced copper supports supporting in turn copper anode plates 2 here shown as in the form of approximately 90° segments. The plates 2 may be made of relatively impure copper. It is not necessary that these plates be made up of copper of a high degree of purity as was the case with the anode

plates used in the prior operation described above. Of course if desired such pure copper plates may be used, but their employment is not important. Thus a considerable saving in anode expense may be effected. These anode plates are suitably supported on the bars 1 by means of wires 3 passing through holes in the plate. The bars 1 are in turn supported by the sides of a lead-lined wooden tank 4 provided with a dished bottom leading to a valved exit which will be described later.

The tank 4 contains an electrolyte bath of copper sulphate and sulphuric acid.

The cathodes consist of a series of rotating copper disks alternating with the anodes 5 and submerged edge-wise in the electrolyte bath.

In order to provide the proper current density passing between the anodes and cathodes, a device which will be described later is provided to control the overflow of the electrolyte and hence its depth. The depth of immersion of the electrodes in the electrolyte determines the current density. For producing a commercial copper powder with this apparatus, I prefer to submerge the cathodes to about one-quarter their depth.

The cathodes are mounted on a shaft 6 supported in bearings 7 at either end of the tank 4, the bearings being suitably insulated from said tank. Mounted on this shaft is a steel sprocket 8 driven by a chain 9 from a smaller sprocket 10 mounted on its shaft 11 supported on said tank. The sprocket 8 is suitably insulated from the shaft 6 by means of an insulated bushing.

A source of current supply, not shown, has its positive pole connected to the anodes through wiring 12 attached to a conducting strip 13 with which the anode bars 1 are in contact. The negative pole is connected by means of wires 14 connected to the bearing block 7.

When current passes the anodes to the rotating cathodes, a thin fluffy deposit or slime of copper is deposited on that portion of the cathode disk 5 which is submerged. The rotation of these disks is preferably continuous and therefore there is a continuous deposition of copper made on both faces thereof. It will be noted that the disks 5 are rotating in clockwise direction as shown in Fig. 1 and therefore the copper is being deposited from the moment that the disk enters the electrolyte on the right side of the tank until it emerges from the left side, then from the left side the copper remains on the disk until it comes to a position just in advance of where it enters the tank. At this point a scraping device is provided shown more clearly in Figs. 1 and 3. The scraping device consists of two troughs 15, the inner edges of which are sharpened to form scrapers 16 and contact each with the near face of one of the cathode disks. These pairs of scrapers are provided for each of the cathode disks in the series. Immediately above each of the troughs 15 are positioned sprays 17 which are connected in a system to be later described for spraying electrolyte on to the copper as it is scraped off. The stream of electrolyte thus carries along the particles of copper with it and the troughs being inclined towards a series of exit pipe 18. The electrolyte with the copper passes through these pipes and into a settling tank 19.

The sprays as noted are part of a circulating system. This circulating system supplies electrolyte for washing and conveying away the copper scraped from the cathodes as noted and includes heating devices for maintaining the electrolyte bath in the tank 4 at about 120° F. which is the preferred temperature for depositing the copper in proper size under the preferred conditions. This system not only includes the sprays 17, pipes 18 and settling tank 19, but also has a receiving tank 20, a heating tank 21 and a pump 22a. The electrolyte bath, the tanks 19, 20, 21 and pump 22a are all connected by piping, so as to permit a continuous flow of electrolyte heated to 120° F. or a selected temperature as desired.

The tank 4 is connected by means of an L-shaped pipe 22 with a pipe 23 leading to an overflow box 24 for the tank 19 which communicates by pipe 25 with receiving tank 20. Situated in the L pipe is a short length of pipe 26 so designed that it projects into the upright portion of the L to give an exit for electrolyte from the tank 4 at the desired height to give a proper current density between the anodes 2 and cathodes 5. If the pipe 26 reaches nearly to the top of the tank 4, the exit through this pipe 26 will insure that the electrolyte bath in tank 4 will rise until it overflows 26 and the depth of liquid then in the tank 4 will be equal to the distance between the bottom of the tank and the top of the pipe 26. By substituting a medium length of pipe somewhat shorter than the one just mentioned, a slightly lower depth of liquid may be maintained and this depth may be varied as desired within the limits of the height of the upright member in the L, by changing the lengths of the pipe 26. Of course the L may have the height of the upright member varied as required.

The receiving tank 19 receives the electrolyte with copper particles suspended therein and when conveyed to this receiving tank, the particles settle out therein, the electrolyte passing off through a pipe 27 into the overflow box 24 and then through the pipe 25 to the receiving tank 20. The pump 22a is connected directly to the receiving tank. It is preferably a motor driven centrifugal pump and is connected by means of piping 28 to deliver into the upper portion of heating tank 21. In this heating tank is a series of steam coils 29 connected with a source of steam and to a discharge not shown. The electrolyte in this tank is heated indirectly by the steam and when heated to approximately 120° F. or other desired temperature passes out through the valved pipe 30 into a pipe 31 which is branched, one branch supplying electrolyte to the sprays 17 and the other branch 32 delivering the heated electrolyte to the tank 4.

The settling tank 19 is provided with a valve 33 at an exit in the bottom thereof, the valve being manually operable by a valve stem 34. When the valve 33 is pulled up, the copper particles with electrolyte pass through a pipe 35 into a delivery pipe 36 from whence it passes into a wheeled filtering car 37 where it is received on a filter tray 38, the electrolyte being sucked off through the suction filter of the car and being later washed by water, care being taken to insure that the copper particles are maintained under the electrolyte or covered with water during the process of removal of the electrolyte, and up to the time the tray 38 is withdrawn and placed in the vacuum dryer. This part of the operation is preferably like that described in the application of Eugene Fitzpatrick, et al. Serial No. 287,033, filed June 20, 1928, Patent No. 1,804,924.

As noted above the anode plates may be of impure copper and where this is the case it will be obvious that impurities therein, which are not deposited by electro-deposition, will fall to the

bottom of tank 4 collecting on the dished surface thereof. In the center of the bottom is a valve 39 seated in an L projection of the pipe 36 provided with a valve stem 40 which may be operated from below to unseat the valve upwardly to allow the impurities in the tank 4 to run out through the pipe 36. These impurities will collect at a rate depending on the purity of the anodes. Tank 4 is cleaned out as often as is necessary and of course these impurities are kept separate from the copper received in the pipe 36 from the settling tank 19. The impurities are not only handled separately in this pipe but they are preferably handled separately in the filter car and in the subsequent drying process.

In carrying out the process of my invention, I prefer to proceed as follows in producing a copper powder. Current is passed from the anodes 2 to the rotating cathode copper disks. Under preferred circumstances the current density at the anodes will average approximately 85 amperes per square foot while the current density at the cathodes will average approximately 250 amperage per square foot. I rotate the copper disks at the rate of about one or less revolutions per minute, the cathodes being submerged to about one-quarter of their depth in the electrolyte to give the desired current density. The anode is immersed substantially entirely. I regulate the current density to the preferred amount for the size of copper particles desired by selecting a pipe 26 of the proper height so that the level of the electrolyte in the tank 4 is sufficient to give the current density desired. The preferred electrolyte contains from about 2.3% to 2.45% of copper, as copper sulphate, and from about 11.3% to 11.4% sulphuric acid. The copper deposited, as the rotating cathodes pass through the tank 4, then passes around through an angle of somewhat less than 180° until it reaches the trough 15 with its scraper where the deposit is scraped off and washed away by electrolyte flowing through the spray 17, from the tank 21. I prefer to maintain the electrolyte at 120° F. and for this purpose a circulating and heating system is provided. The electrolyte flowing from the tank 4 passes downwardly through the overflow box 24 of the settling tank and thence it passes into the receiving tank. The copper particles washed away by the spray 17 flow into the pipes 18 and thence into the settling tank where the copper settles out and may be removed when collected in sufficient quantity from the settling tank 19. The excess electrolyte passes into the settling tank overflow box and then into the receiving tank 20. From the latter the pump 22*a* pumps the electrolyte up to the heating tanks 21 where it is heated and then allowed to flow by gravity to supply electrolyte to the cell 4 and spray 17. The settling tank is dumped periodically into the filter 37, the copper powder being washed on the filter, the washing being preferably done with boiled water at a temperature somewhat below 120° F. in order to prevent oxidation. It is then transferred to the vacuum dryer and dried, preferably in accordance with the application of Fitzpatrick et al. mentioned above.

The impurities settling out from the anodes are collected by dumping the electrolytic cell tank and allowing the impurities to run into car 37 where they are washed and then dried in a vacuum dryer.

All of the copper powder produced as above in accordance with my invention, was removed from the vacuum dryer and found to pass through a 200 mesh screen. The resultant product averaged 99.6% purity. In some instances it was as high as 99.73% pure. The theoretical amount of finely divided copper in pounds that can be produced by 90 hours using a current of about 890 amperes, is equal to 210 pounds. The ampere efficiency secured by operation of my invention, I have found to be

$$\frac{178 \text{ pounds}}{210 \text{ pounds}} = \text{about } 85\%$$

The apparatus is of simple construction. The current density maintained by it varies little, if any, during the operation of the apparatus and consequently the particle size of the resultant copper or other metal is substantially uniform. Since the cathodes are continuously rotating and are being sprayed over their whole surface by the electrolyte, the difficulty of having oxidation at the liquid level of the electrolyte tending to eat away the cathode as in the case of rods used in the old practice, is eliminated. The apparatus and process operates with high efficiency. Furthermore impure anodes may be used thus saving the expense of these electrodes.

As pointed out above other metals than copper which are susceptible to electro-deposition in the manner indicated, may be employed in the apparatus described. In addition varying current densities may be employed as required or preferred. It will, of course, be understood that I do not restrict myself to anodes of any particular impurity, but I find it desirable to use a commercial copper as anodes containing approximately a total of .3% of solid impurities as gold, silver, lead, nickel, cobalt, antimony and arsenic.

Having thus described my invention with particularity with reference to the preferred method of carrying out the same, it will be obvious to those skilled in the art, after understanding my invention, that various changes and modifications may be made therein without departing from the spirit and scope of my invention, and I aim in the appended claims to cover such changes and modifications as are within the scope of the invention.

What is claimed as new and desired to secure by Letters Patent is:

1. In apparatus for making metal powder, in combination, an electrolyte bath, an anode and a movable cathode therein, means for passing an electric current from the anode through the electrolyte to the cathode, means for removing the metal deposit formed on the cathode without passing it through the bath, and means for conveying the deposit away in a non-oxidizing medium.

2. In apparatus for making metal powder, in combination, an electrolyte bath, an anode and a rotatable cathode therein, means for passing an electric current from the anode through the electrolyte to the cathode, means for scraping off the metal deposited on the cathode while being rotated, and means for conveying the deposit away in a non-oxidizing medium without passing it through the bath.

3. In apparatus for making metal powder, in combination, a soluble anode containing impurities therein, a moving cathode, an electrolyte bath forming a circuit between said anode and cathode, means for scraping off and keeping separate from said bath and impurities therein the metal deposit formed on said cathode, and means for excluding atmospheric air from the deposit

to prevent oxidation and consequent discoloration of the powder.

4. In apparatus for making metal powder, in combination, an electrolyte bath, an anode and a rotatable cathode therein, means for passing an electric current from the anode through the electrolyte to the cathode, means for scraping off the metal deposited on the cathode while being rotated, and means for conveying the deposit away in a stream of electrolyte without passing it through the bath.

5. In apparatus for making metal powder, in combination, a series of anodes, a series of rotatable cathodes placed between said anodes, an electrolyte bath in which said anodes and cathodes are immersed, a source of current supply connected to said anodes and cathodes, a scraping device outside said bath for scraping off copper deposited on said cathodes, means also located outside of said bath for receiving the deposit, and means for protecting the deposit from oxidation by exposure to air.

6. In apparatus for making copper powder, in combination, an electrolyte bath, a series of soluble relatively impure copper anodes spaced apart immersed therein, a series of rotatable copper cathode disks partially immersed in said bath and placed between said copper anodes, a source of current supply connected to said anodes and cathodes, said disks receiving copper deposited thereon by said current and conveying it by rotation to a point outside said bath, a scraper outside said bath for scraping off copper deposited on said copper disks, and means for conveying the deposit away in a non-oxidizing medium.

7. In apparatus for making metal powder, in combination, anodes and cathodes, an electrolyte bath in which said anodes and cathodes are submerged, a source of current supply connected to said anodes and cathodes, means for removing the metal deposit from the cathodes, and a circulating system for supplying and withdrawing electrolyte at an elevated temperature, including means for spraying a stream of electrolyte on the deposit after it has been removed from the cathodes to thereby protect the deposit from oxidation by exposure to air.

8. In apparatus for making copper powder, in combination, a series of relatively impure copper anodes spaced in an electrolyte bath containing copper sulphate and sulphuric acid, a series of rotatable cathode copper disks placed between said anodes and submerged edgewise to about one-quarter their depth in said bath, a source of current supply connected to said anodes and said cathodes, a scraper for scraping off copper deposited on said cathode disks, a circulating system for supplying electrolyte for washing and conveying away said powder scraped from said cathodes and for maintaining said bath at about 120° F. comprising a settling tank, a receiving tank, a heating tank and a pump, said bath, tanks and pump being connected to one another to provide a flow of electrolyte therethrough, an adjustable overflow device in the connection between said bath and said receiving tank to adjust the height of electrolyte in said bath and thereby the density of current passing to said cathodes, a trough connected to said scraper placed beneath said washing device and conveying powder washed off said cathode thereby through a connection to said settling tank, an overflow from said settling tank to said receiving tank, said pump being connected to said receiving tank and delivering electrolyte to said heating tank located above said bath, a device for heating the electrolyte in said tank to about 120° F., said electrolyte flowing by gravity to said washing device and to said bath at the desired temperature, a suction filter car, and valved connections between said settling tank in the bottom of said bath to said car, whereby upon opening the valve in said settling tank copper powder passing into said filter car may be washed therein for subsequent drying, or the impurities in the bottom of said bath may be conducted into said car and similarly treated.

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