

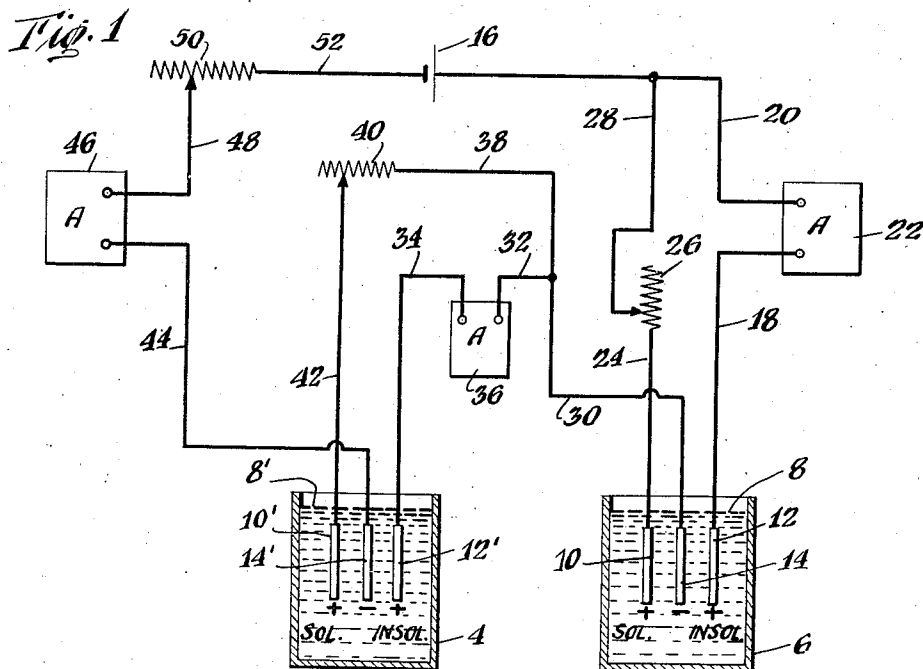
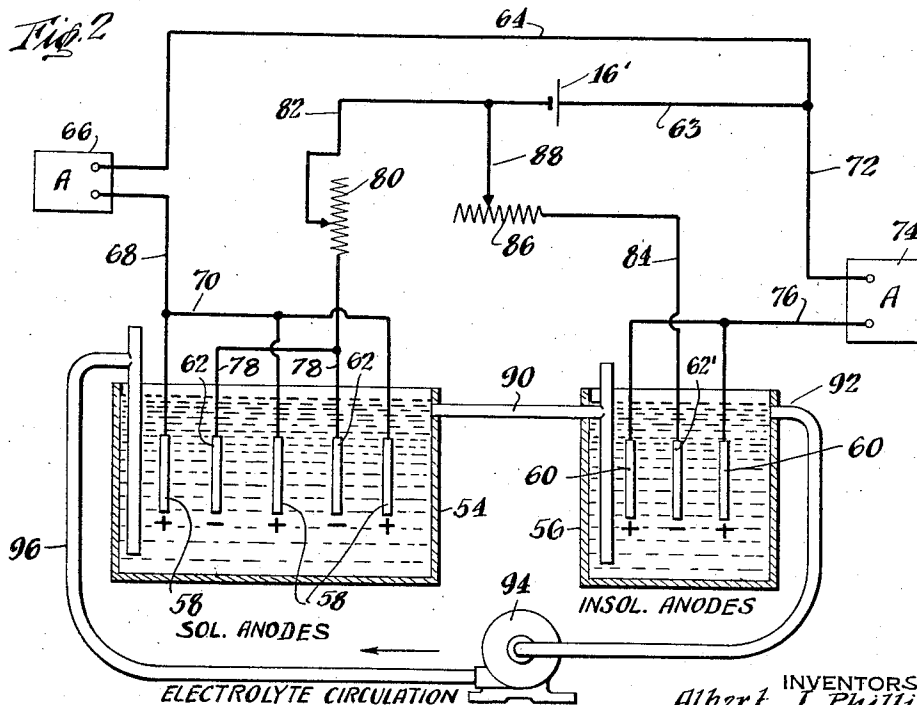
Sept. 12, 1944.

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2,358,029

PROCESS OF ELECTRODEPOSITING INDIUM

Filed March 2, 1940



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UNITED STATES PATENT OFFICE

2,358,029

PROCESS OF ELECTRODEPOSITING
INDIUM

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Application March 2, 1940, Serial No. 321,839

3 Claims. (Cl. 204-45)

The present invention deals with certain improvements in the electrodeposition of indium.

It is known in the art to electroplate indium from cyanide baths or chloride baths with the use of suitable addition agents, it being considered essential for the production of adherent deposits of indium that the baths be either a cyanide or chloride bath. However, in the operation of such baths, there are difficulties, owing, for instance to the instability of the cyanide electrolyte, and anode corrosion due to chlorine evolution, from the chloride electrolytes.

In the prior art operations for the electrodeposition of indium, either in refining or plating operations, care has been taken to avoid sulphate electrolytes owing to the difficulty or impossibility of obtaining uniform deposits of indium from such sulphate electrolytes, the deposit of indium, if formed at all, being of a spongy and non-adherent character.

The present invention provides an improved process for electrodepositing indium either in refining or plating operations, wherein a smooth, adherent deposition of indium is effected by the electrolysis of a solution of indium sulphate, thereby avoiding the difficulties incident to the use of cyanide or chloride electrolytes.

Apparently, the difficulty of producing satisfactory deposits of indium from the electrolysis of sulphate electrolytes resides in the fact that during the electrolysis the acidity of the sulphate solution changes very rapidly, this condition having been observed to take place during the investigations leading up to the present process, the early stages of the investigation producing the same spongy deposit of indium which were noted in the prior art in connection with the electrolysis of indium sulphate solution.

It has been found, however, in the investigations which resulted in the present improved process, that very satisfactory deposits of indium are obtainable from indium sulphate electrolytes, even without the use of addition agents or buffers, if the acidity of the electrolyte is maintained between carefully controlled limits, under properly controlled operating conditions, there being obtained the dense crystalline and adherent deposit of indium which has been mentioned above, this deposit being obtained easily and without complication of procedure if the conditions of electrolysis are maintained properly.

One of the important factors in the practicing of the present improved process is to maintain

a controlled pH value of the sulphate bath between definite limits, which limits are found in practice to be in the range of from about pH 1.5 and pH 3.4, it being found that the maintenance of this controlled pH value results in the production of the desired dense indium deposit.

In practice it is found to be an essential factor to effect this control of the acidity of the electrolyte. This control may be effected by the use either of soluble or insoluble anodes, preferably by a combination thereof. If soluble indium anodes are employed, it will be found to be necessary to control the acidity of the electrolyte by additions of sulphuric acid as the process proceeds and also by withdrawing periodically a portion of the electrolyte and replacing with water to maintain the indium-ion concentration at its original value. Where insoluble anodes are employed, it may be desirable to control the acidity of the electrolyte between proper limits by withdrawing a portion thereof as the electrolysis proceeds, adding to the withdrawn portion a suitable quantity of indium hydroxide which quantity is determined by the pH value of the solution, so that when the thus-treated withdrawn portion is reintroduced into the cell, the proper pH value will be maintained. The withdrawn portion of the electrolyte may be filtered after this treatment with the indium hydroxide, and the volume made up with any necessary addition of water.

Because of the necessity of treating the electrolyte in such a manner as has been described above where either insoluble or soluble anodes are used for the electrolysis, it is preferred to carry out the process by using both insoluble and soluble anodes. Such anodes may be employed in the same cell, or the soluble and insoluble anodes may be placed in separate cells with an intercell circulation of the electrolyte being maintained. The soluble anodes of course are formed from metallic indium, the insoluble anodes being, suitably, platinum.

The indium content of the electrolyte may vary between wide limits, the process having been operated successfully with a range of from 5 grams per liter of indium to 130 grams per liter of indium, the pH value of the electrolyte of course being controlled in accordance with the concentration of indium in the solution, the higher concentrations of indium being operated in connection with the lower limits of the pH control, and low concentrations of indium being operated with the higher pH values, it being important to maintain sufficient acidity in the

electrolyte to prevent precipitation of the indium by hydrolysis.

It is desirable to maintain the electrolyte in circulation, by stirring in the cell, or by extra-cell circulation. The electrolysis may proceed at room temperatures, although slightly elevated temperatures seem to give slightly better deposits in some cases.

The electrolysis is carried out with a cathode current density of a wide range, the process having been carried out with cathode current density ranging all the way from 2 amps./sq. ft. to 40 amps./sq. ft., it being found in practice, however, that a cathode current density of about 2.4 amps./sq. ft. for refining, and about 30 amps./sq. ft. for plating gives the best results.

The anode current density also is variable. In refining operations, the current density depends on the purity of the anode, but is kept as low as possible, the best range being from approximately 2 to 4 amps./sq. ft. In plating operations, with no thought to purification of the deposit, the anode current density may be very high, in practice, for example, 40 amps./sq. ft. or higher.

The cell employed is an undivided cell, and the soluble anodes may be enclosed in a bag if desired, or the operation may be conducted without a bag around the anodes. In the latter instance the electrolyte must be filtered from time to time to remove slimes from the bottom of the cell.

The usual arrangement of electrodes is employed.

The accompanying drawing illustrates typical simple cell installations which may be used in carrying out the present improved process, it being understood, however, that the particular illustrated showings may be modified quite widely without effecting the electrolysis proceeding in the cells, the views of the drawing showing merely examples of the simplest arrangements of apparatus suitable for carrying out the process.

In the drawing, Fig. 1 represents a type of wiring diagram suitable for use with a combination of soluble and insoluble anodes in the same cell, there being a plurality of cells shown in series.

Fig. 2 is a view similar to Fig. 1, wherein the soluble and insoluble anodes are grouped in separate cells, there being provided inter-cell circulation for the electrolyte.

Referring more particularly to the drawing, and first to Fig. 1, two electrolytic cells 4 and 6 are shown, which cells contain an electrolyte 8 of indium sulphate. Either cell 4 or 6 contains also a soluble anode 10 and an insoluble anode 12, the anodes 10 and 12 having a cathode 14 placed between them.

These electrodes of course are of suitable shapes and sizes and their number may be varied at will. The reference characters in cell 4 are primed to avoid confusion.

A suitable electrolyzing current is supplied to the cells from a source 16 of direct current, the anodes of cell 6 being connected to the positive side of the current source 16, the insoluble anode 12 of cell 6 being connected to the positive side of the current source by current leads 18 and 20 through an ammeter 22, while the soluble anode 10 is connected to the positive side of the current source through lead 24, adjustable resistance 26 and lead 28.

The cathode 14 of cell 6 is connected by suitable leads 30, 32, 34 to the insoluble anode 12' of cell 4, through an ammeter 36, and also to the soluble

anode 10' through leads 30, 38, adjustable resistance 40 and lead 42.

The cathode 14' of cell 4 is connected by lead 44 through ammeter 46, lead 48, adjustable resistance 50 and lead 52 to the return side of the current supply.

Ammeters 22 and 36 indicate the current through the insoluble anodes of cells 6 and 4, respectively, and ammeter 46 indicates total amperage in the system. Resistances 26 and 40 are of the comparable magnitude, and that of resistance 50 depends upon the voltage drop across the power source.

In Fig. 2, there is indicated an arrangement wherein the soluble anodes and insoluble anodes are in separate cells, with inter-cell circulation means. In the system of Fig. 2, there are shown two cells 54 and 56, the cell 54 receiving the soluble anodes 58, and the cell 56 receiving the insoluble anodes 60. Cathodes 62, 62' are positioned between the anodes.

A suitable source of direct current 16' has its positive side connected to the soluble anodes 58 in cell 54 and to the insoluble anodes 60 of cell 56. The connections to the soluble anodes are made through leads 63, 64, ammeter 66 and leads 68 and 70, the latter being branched as required for requisite anodic connections. The positive side of the current source is connected also with the insoluble anodes 60 through leads 63, 72, ammeter 74 and lead 76, which is branched as required for requisite anodic connections.

The cathodes 62 in the cell 54 are connected by leads 78, adjustable resistance 80 and lead 82 to the return side of the current source, and the cathodes 62' in the cell 56 are connected to the return side of the current source through lead 84, adjustable resistance 86, and lead 88.

The cells are interconnected as shown at 90 and electrolyte is withdrawn from cell 56 through pipe 92 by pump 94 and delivered by the pump 94 into cell 54 by way of pipe 96.

In Fig. 2, the ammeter 66 measures total current in the cell 54 and the ammeter 74 measures the total current in cell 56.

Obviously, arrangements of equipment other than these herein specifically illustrated may be employed, with comparable electrolytic results being obtainable. Thus, it is possible, easily, to form the electrical circuits in such a way as to require the use of only one, or at most two, ammeters, instead of the larger number shown in the illustrated circuits.

The process is illustrated by the following specific example, which illustrates the conditions of electrolysis in a typical run: Two cells were used, the electrolyte being circulated between them, one of the cells containing soluble anodes of indium, the other cell containing insoluble anodes composed of platinum.

In refining operation, in the soluble anode cell there was maintained a current density of 2.4 amps./sq. ft. at both anode and cathode. The voltage drop was from 0.45 to 0.6 volt.

In the insoluble anode cell, the current strength is 0.5 amp. with a cathode current density maintained at 2.4 amps./sq. ft., with voltage drop of from 3.1 to 3.5 volts.

The system was run for 10 days at room temperature at a pH of from 2.1-2.3, a compact crystalline deposit being obtained. The current efficiency was from 95 to 100%, and the indium content of the electrolyte was maintained at 18.2 grams per liter of indium as indium sulphate.

In plating, a pH value the same as above was

maintained by the same ratio of soluble to insoluble anode current strengths. With a cathode current density of 30 amps./sq. ft., a smooth continuous plate of indium, 0.005" thick was put on a lead base bearing alloy as cathode and on cadmium-nickel alloy cathode at a current efficiency of about 65%. The electrolyte was a solution of indium sulphate maintained at a controlled pH of from 2.1-2.3.

If desired, one or more of the alkali sulphates such as sodium sulphate or ammonium sulphate, in effective amounts, say 25 gms./liter may be added to the electrolyte for reducing the resistance thereof, with no bad effects on the deposit; also insoluble colloidal organic addition agents, such as glue or gelatin may be incorporated in the electrolyte for facilitating smoothness in heavy deposits.

What is claimed is:

1. A process of electrodepositing indium metal on a cathode surface which comprises passing electric current through a prepared electrolyte solution consisting of an aqueous solution of indium sulphate and sulphuric acid, said solution containing from 5 to 130 grams of indium per liter of electrolyte, using soluble indium anodes and insoluble anodes with cathode electrodes under pH controlled conditions in order to form a dense, coherent crystalline deposit of metallic indium on the cathode, and wherein the pH value of said electrolyte is maintained between 1.5 and 3.4 during electrolysis by means of said soluble and insoluble anodes to cause the deposition of a smooth adherent deposit of indium on the surface of the cathode.

2. A process of electrodepositing indium metal on a cathode surface which comprises passing electric current through a prepared electrolyte solution consisting of an aqueous solution of indium sulphate and sulphuric acid, which solution contains about 18.2 grams of indium per liter, using soluble indium anodes and insoluble anodes with cathode electrodes under pH controlled conditions so as to form a dense crystalline deposit of indium on the cathode, said pH value being controlled within the range of from 2.1 to 2.3, during electrolysis by means of said soluble and insoluble anodes to cause the formation of a smooth, continuous and adherent deposit of indium metal on said cathode surface.

3. A process of electrodepositing indium metal on a cathode surface which comprises providing an electrolyte solution composed of indium sulphate dissolved in aqueous sulphuric acid and wherein the indium content of said electrolyte solution approximates about 18.2 grams per liter, passing electric current through said solutions under pH controlled conditions using soluble indium anodes and insoluble anodes and cathode electrodes with a cathode current density of between 2 and 40 amperes/sq. ft. to cause the indium to be deposited out on the cathode, and during said deposition maintaining the pH value of the indium sulphate solution between 1.5 and 3.4 by means of said soluble and insoluble anodes whereby indium metal is deposited on said cathode surface as a firm coherent crystalline deposit of metallic indium.

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