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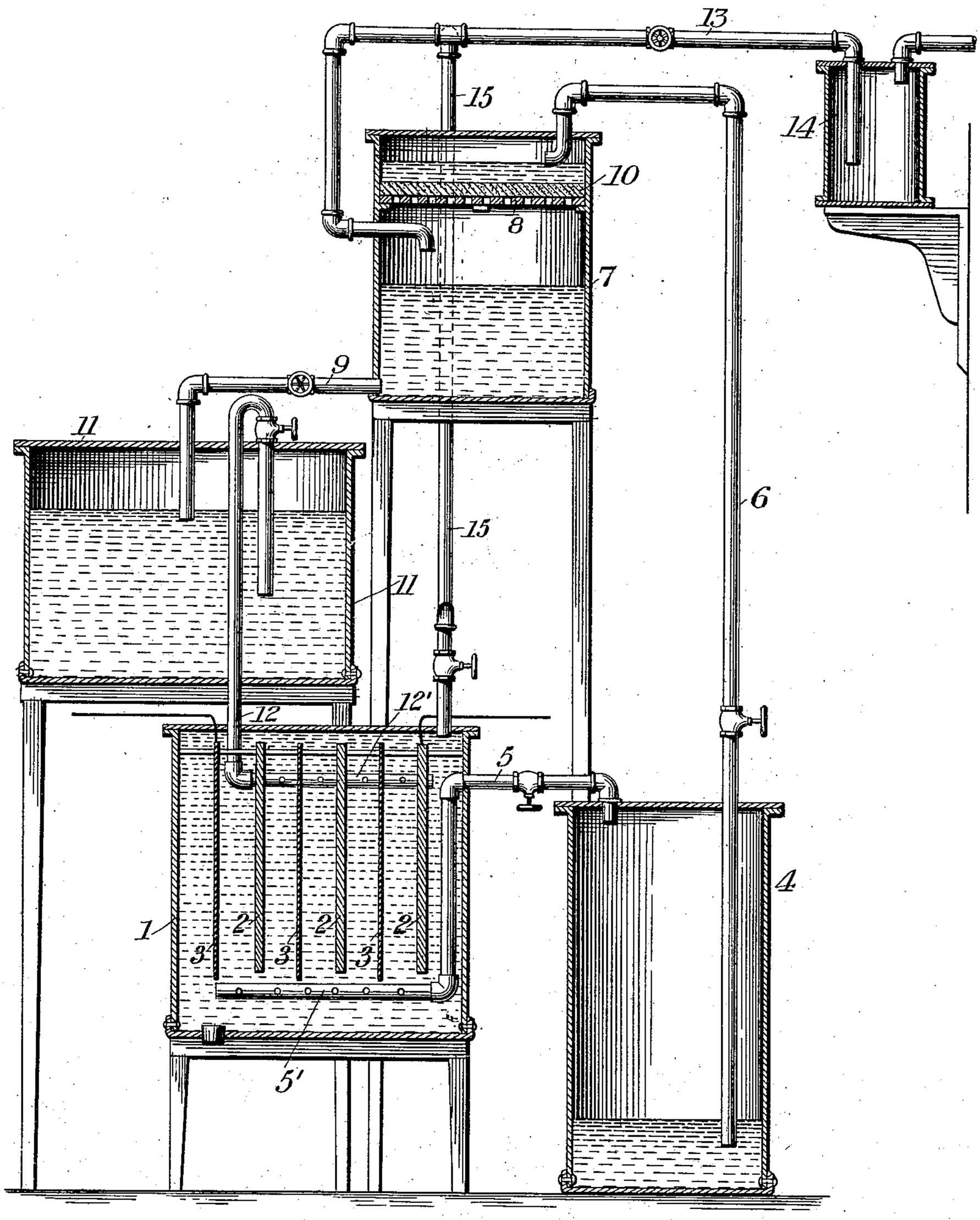
W. McA. JOHNSON.

PROCESS OF ELECTRODEPOSITING METALS.

APPLICATION FILED JAN. 13, 1903.

NO MODEL.



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UNITED STATES PATENT OFFICE.

WOOLSEY McA. JOHNSON, OF HARTFORD, CONNECTICUT.

PROCESS OF ELECTRODEPOSITING METALS.

SPECIFICATION forming part of Letters Patent No. 742,442, dated October 27, 1903.

Application filed January 13, 1903. Serial No. 138,857. (No specimens.)

To all whom it may concern:

Be it known that I, WOOLSEY McA. JOHNSON, a citizen of the United States, residing at Hartford, in the county of Hartford and State of Connecticut, have invented certain new and useful Improvements in Processes of Electrodepositing Metals, of which the following is a specification.

This invention relates to the electrodeposition of metals under such conditions as will yield a deposit of improved character.

In the deposition by means of the electric current of such metals as are electropositive to hydrogen, of which metals nickel may be taken as an example, a certain quantity of hydrogen, depending upon the current density used, is liberated at the cathode at the same time that the metal is deposited thereon. This hydrogen is in part occluded by the deposit, in part adheres thereto, and is in part dissolved by the electrolyte or otherwise held therein. I have discovered that this gas in some or all of the conditions above stated exerts an injurious effect upon the tenacity, density, and other characteristics of the electrodeposited metal. Furthermore, in the electrodeposition of metals, such as copper, which are electronegative to hydrogen, an evolution of gas may also occur at the cathode, especially when a certain limit of current density is exceeded. Such metals also are injuriously affected by the gas. Furthermore, all liquids which are capable of serving as electrolytes contain under normal conditions considerable quantities of dissolved or otherwise retained gases, which may appear in part in the form of bubbles when the electrolyte is subjected to a slight rise of temperature. These gases also I have found to affect the deposited metal in an injurious manner.

My invention consists in performing the depositing operation under such conditions and with such materials as will avoid the deleterious action of such gases.

I have found that if the electrolyte is substantially freed from gases before the metal is deposited therefrom, and especially if it be maintained substantially free from gases during the plating operation, the quality of the separated metal is greatly improved, as evidenced by its increased density, hardness, and toughness, by its smooth surface, and by other physical and chemical characteristics. As a suitable and convenient means for maintaining the electrolyte in this condition I make use of a vacuum or such diminished pressure of air as will to a sufficient extent accomplish the result. Under diminished atmospheric pressure the gases held or liberated within the electrolyte quickly escape therefrom, and the solution is therefore maintained in a substantially gas-free condition. I may also maintain the electrolyte in circulation between the electrodes, and, if desired, I may cause it to circulate through a suitable filter capable of removing and retaining any solid particles. I find that the diminished air-pressure above referred to constitutes a convenient means for accomplishing this circulation, and to this end I prefer to exhaust the air from beneath a filter contained in a vessel or vessels situated above and suitably connected with the electrolytic tank, thereby transferring portions of the electrolyte through the filter. Such portions are then permitted to flow again to the electrolytic tank. By providing a plurality of filtering vessels and using them alternately the flow of the electrolyte may be made continuous.

For a clear understanding of the invention reference is made to the accompanying drawing, which shows one form of my apparatus in vertical section, and wherein—

1 indicates an electrolytic tank, and 2 3 anodes and cathodes mounted therein and suitably connected to a source of electric current.

4 is a closed overflow-tank connected by a valved pipe 5 with the electrolytic tank and by a valved pipe 6 with the filtering-tank 7. Said tank 7 contains a distributing-plate 8 for the electrolyte, upon which is preferably placed a suitable filter 10. Said tank 7 communicates through a valved pipe 9 with a reservoir 11, from which the electrolyte is discharged through a pipe 12 to the electrolytic cell 1. Said pipe 12 is preferably arranged, as shown, to draw the electrolyte from beneath the surface, and said tank 11 is preferably closed in order that the air above the

surface of the electrolyte may be maintained at diminished pressure. Preferably pipes 5 and 12 are provided within the electrolytic tank with perforated sections 5′ and 12′, extending transversely of the electrodes and serving to provide a distributed flow of the electrolyte along their surfaces.

The filtering-tank 7 is hermetically closed and communicates through a valved pipe 13 with a vacuum-pump or other device for removing gases. (Not shown.) A trap 14 for collecting any liquid drawn over through pipe 13 may be interposed, as shown in the vacuum-line. Said pipe 13 enters the tank 7 below the filtering layer 10, but above the normal level of the electrolyte. The electrolytic tank 1 is shown as closed and provided with a valved pipe 15, leading to the vacuum-line 13.

In operation the electrolyte is permitted to flow from tank 7 through pipe 9 into a tank 11, thence through pipes 12 and 12′, the electrolytic vat 1, and pipes 5′ and 5 to the tank 4. By the operation of the pump the liquid in tank 4 is raised through pipe 6, discharging above the filter in tank 7. The liquid then flows through the filter and several tanks, as before specified. In this construction the flow of the electrolyte is necessarily intermittent; but it will be readily understood that by providing a plurality of tanks 7 with suitable connections thereto and by using such tanks in alternation the flow may be rendered continuous. It will thus be seen that portions of the electrolyte are submitted to the action of diminished air-pressure serving, as above stated, to withdraw therefrom the contained gases. The electrolyte reaches the electrolytic tank 1 in a virtually gas-free condition, and the flow through such tank should be sufficient to transfer the electrolyte to the filtering-tank 7 with sufficient rapidity to maintain the gas content low enough to avoid injury to the deposit. The electrolytic tank 1 may be open to the atmosphere. Preferably, however, it is closed and communicates with the exhausting device, as shown, in order that the gases liberated by the electrolysis may be rapidly withdrawn and the electrolyte maintained at all times in a substantially gas-free condition.

It is obvious that the overflow-tank 4 may be open to the atmosphere without interfering in any manner with the operation of my device. I prefer, however, that the several tanks be closed and placed in communication with the vacuum-pump, inasmuch as additional liquid-surface for the liberation of gases is thereby provided.

While I have described the electrolyte as being subjected during the electroplating to such conditions as will determine the expulsion or exhaustion of gases, I find that good results may also be attained by a preliminary treatment of the liquid to expel the gases, the electroplating then proceeding under the normal pressure. My preferred procedure is, however, as above described, the electrolyte being circulated and subjected both within and without the electrolytic field to the action of diminished pressure. I may also heat the liquid to any desired temperature either within the electrolytic tank or in a vessel or conduit external thereto in order to aid in the explusion of the gases. I am aware that electrolytic operations of various kinds have been conducted under diminished air-pressure with the object of reducing polarization or of removing a product of the electrolysis. I am not aware, however, that such diminished pressure has been employed to withdraw gases from the electrolyte for the purpose of improving the character of electrodeposited metal or that the deposition of metal has been effected under these conditions.

I claim—

1. The herein-described method which consists in subjecting the electrolyte in the neighborhood of the cathode to such pressure as will remove contained gases, and electrodepositing a metal therefrom in reguline form.

2. The herein-described method which consists in subjecting the electrolyte as a whole to such pressure as will remove contained gases, and electrodepositing a metal therefrom in reguline form.

3. The herein-described method which consists in subjecting the electrolyte in the neighborhood of the cathode to such pressure as will remove contained gases and simultaneously electrodepositing a metal therefrom in reguline form.

4. The herein-described method which consists in first subjecting an electrolyte to such pressure as will remove contained gases, then electrodepositing a metal therefrom in reguline form, and maintaining the electrolyte under such pressure as will remove the gases from the region of deposition.

5. The herein-described method which consists in circulating an electrolyte between an electrolytic tank and an external vessel, maintaining a diminished atmospheric pressure in said tank and vessel, and passing an electric current through said electrolyte.

6. The herein-described method which consists in circulating an electrolyte between an electrolytic tank and a filtering vessel, maintaining a diminished pressure in said tank and filtering vessel, and passing an electric current through said electrolyte.

7. The herein-described method which consists in removing contained gas from an electrolyte, and depositing a metal from such gas-free electrolyte in reguline form.

8. The herein-described method which consists in removing contained gas from an electrolyte, electrodepositing a metal therefrom in reguline form, and maintaining the elec-

trolyte substantially free from dissolved gases in the region of such deposition.

9. The herein-described method which consists in electrodepositing a metal in reguline form from a substantially gas-free electrolyte.

10. The herein-described method which consists in circulating an electrolyte past a region of deposition, removing from such electrolyte the contained gases, and depositing a metal from such electrolyte in reguline form.

In testimony whereof I affix my signature in presence of two witnesses.

WOOLSEY McA. JOHNSON.

Witnesses:

HENRY H. PEASE,

CHARLES M. STARKWEATHER.