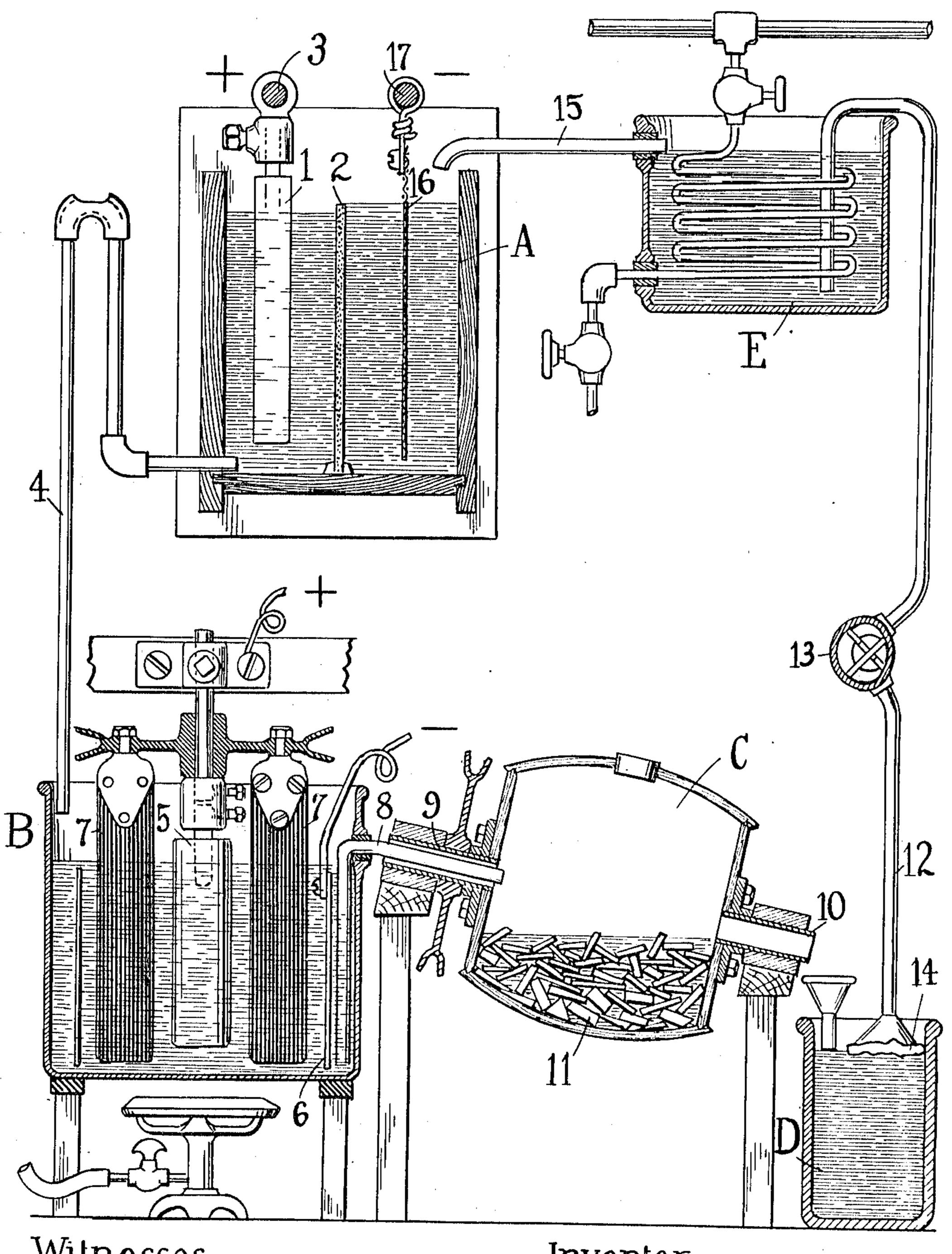
## P. F. COWING.

PROCESS OF PRODUCING ALLOYS AND THE SEPARATION OF METALS. APPLICATION FILED NOV. 11, 1909.

969,773.

Patented Sept. 13, 1910.



Witnesses:

Samuel W. Balak. Frank O. Cole.

Inventor,

Percy Foote Cowing, by Ling, fr., Attorney.

## UNITED STATES PATENT OFFICE.

PERCY FOOTE COWING, OF NEW YORK, N. Y.

PROCESS OF PRODUCING ALLOYS AND THE SEPARATION OF METALS.

969,773.

Specification of Letters Patent. Patented Sept. 13, 1910.

Application filed November 11, 1909. Serial No. 527,407.

To all whom it may concern:

Be it known that I, Percy Foote Cowing, a citizen of the United States of America, and a resident of the city of New York, county of New York, and State of New York, have invented certain new and useful Improvements in Processes for Producing Alloys and the Separation of Metals, of which the following is a specification.

and relates to the electrolytic treatment of alloys containing principally copper and nickel, and in certain cases iron, the object being to separate the copper and to recover nickel and iron economically and by simple operations. The nickel and iron are obtained in the form of a compact thick deposit on the cathode.

In carrying out my process the original alloy is placed as an anode in an electrolytic bath, which, upon the passage of the current has the property of dissolving substantially all of the metals of the original alloy. That portion of the electrolyte forming the resultant solution is then treated in such manner as to replace the copper by iron, or nickel and iron, and nickel and iron are thereafter electrolytically deposited.

Though I do not limit myself to the use thereof, I have found that a warm, rather concentrated substantially neutral aqueous solution of nickel chlorid is a suitable initial electrolyte for carrying out the process. The very soluble nickel chlorid dissolved in water as described forms, especially when hot, an electrolyte of low electrical resistance and permits the use of high current densities. If the precautions herein pointed out are observed, such an electrolyte adjusts itself during the operation of the process to the particular original alloy under treatment, as will be readily understood.

Well known instrumentalities can be em-

Well known instrumentalities can be employed in performing my process, and for the purposes of illustration, I have shown one form of apparatus by which the process can be worked, but without intending to be confined thereto.

In the accompanying sheet of drawings, which forms a part of this application, the figure is a cross-section through the several instrumentalities constituting the system.

The first vat A comprises an anode compartment and a cathode compartment, wherein the copper bearing alloy 1 to be

treated is dissolved as the anode, and nickel and iron are deposited on the cathode. The anode and cathode compartments are separated by a diaphragm 2. The rest of the apparatus is for modifying the copper-con- 80 taining solution which is withdrawn from the anode compartment of this vat A and for discharging it, after proper treatment for the removal of the copper therefrom, into the cathode compartment thereof. The 65 anode and cathode and the electrolytes in the two compartments of this vat constitute a divided electrolytic cell in which the alloy to be treated is dissolved and the desired metal or metals obtained. The dividing 70 diaphragm of the vat is of that type which is sufficiently non-porous to prevent the mixing of the solutions on either side thereof to any appreciable degree, but at the same time has sufficient porosity to allow the electric 75 current to be conducted therethrough by the electrolyte in its pores without too greatly increasing the resistance. The original alloy is connected as the anode to a positive bus-bar 3. When the electric cur- 80 rent is passing, the original alloy goes into solution in the electrolyte in the anode compartment of the vat A. The solution thus formed is carried from the bottom of this compartment through a conduit 4 into a 85 second electrolytic vat B in which selective deposition is carried on. The anode 5 in this vat is composed of nickel or iron or both, and the cathode 6 may be composed of copper; but this is not essential. The 90 electrolyte is preferably agitated by stirrers 7 and may be heated. By maintaining a sufficiently low impressed voltage between the anode and cathode, the copper-containing solution from the anode compartment 95 of the first vat A in its passage through the second vat B is caused to deposit copper selectively upon the cathode 6, an equivalent amount of the anode 5 going into solution to replace the copper. It is not ex- 100 pected that all of the copper in the solution be so replaced. It is obvious that a series of vats for selective deposition of copper may be used. The solution is withdrawn from the vat for selective deposition, or 105 from the last of these vats if more than one is used, through a conduit 8 which communicates with the interior of a receptacle or series of receptacles C wherein the copper still remaining in solution is removed 110

by cementation on iron. It is here shown as a tumbling barrel mounted on an inclined axis by means of hollow trunnions 9 and 10, the conduit entering through the 5 upper trunnion. This tumbling barrel contains loose pieces or fragments of iron 11 which it is desired to substitute for the copper remaining in solution after leaving the selective deposition vat. While the substi-10 tution is herein being effected by cementation, the tumbling action is of advantage as it dislodges some of the cemented copper from the iron fragments in the receptacle and prevents their adherence to each other 15 and, by agitation, continually brings fresh solution into contact with the fragments. As already indicated a series of these cementation receptacles may be used. Sufficient time is allowed during the passage of the solution through the vat for selective deposition B and the cementation receptacle C to permit the replacement of the copper. When this replacement has taken place sufficiently, the solution is discharged from 25 the cementation receptacle, or the last of these if more than one is used, through the lower trunnion into a settling tank D, in which any undissolved material carried over mechanically is allowed to settle. The so-30 lution now freed from the salts of copper, and containing the salts of iron, or iron and nickel, substituted therefor, is conveyed from the settling tank to a heating tank E through a conduit 12 by any suitable means, 35 a pump 13 being illustrated. A filter 14 can be introduced between the settling tank and the heating tank, if desired. This solution is then conveyed through a conduit 15 from the heating tank to the cathode compartment 40 of the first vat A, the temperature of the solution being raised to a sufficient degree in the heating tank or in the vat A to maintain in the cathode compartment that temperature of the electrolyte at which the 45 electrolytic process is most efficiently carried on. The bath in this cathode compartment contains nickel and iron in solution, A cathode 16 is immersed therein upon which metals are deposited from the solu-50 tion. This cathode is suspended from a negative bus-bar 17. When the desired amount of metal has been deposited upon the cathode, the cathode is removed. The solution in the cathode compartment is al-55 lowed to flow over the diaphragm 2 into the anode compartment, thereby completing the circuit of flow. If desired the electrolytes in the anode and cathode compartments of vat A may be agitated by any well known do means. The deposit, or the cathode and deposit, constitute the product which my process is designed to secure. By depositing predetermined weights of nickel, or nickel and iron, upon a cathode of known weight 65 and material, as iron, the relative weights

of these metals in the cathode and its deposit may be varied as desired.

In carrying out my process, under certain conditions where the amount of copper in the original alloy is not excessive and it is 70 not desired to control the percentage of iron in the final product, the second electrolytic vat B may be omitted. The electrolyte is then discharged from the anode compartment of the first vat A directly into the cem- 75 entation receptacle where the copper in solu-

tion is replaced by iron.

Throughout the process the iron and copper salts are formed and should be mainained as far as possible in their reduced 80 condition as ferrous and cuprous salts. This is very advantageous as the copper is then in the best condition for cementation, requiring the smallest amount of iron for its replacement, and the iron is in the best con- 85 dition for electro-deposition. As no insoluble anodes are used, the electrolyte remains at all times electro-chemically balanced. By this I mean, that whenever a metal is removed, either by electro-deposi- 90 tion or cementation, an equivalent of some metal or metals goes into solution in its place; hence no energy is wasted in gas generation, nor is the substantially neutral condition of the electrolyte disturbed. There 95 fore as the electrolyte is not destroyed and adjusts itself to different original alloys, the process can be carried on indefinitely by merely supplying fresh anodes and cathodes and iron for cementation. When using the 100 chlorid electrolyte referred to, the solution withdrawn from the anode compartment of the vat A is or becomes a mixture of nickel chlorid, ferrous chlorid and cuprous chlorid, and after the replacement of the copper, it 105 becomes a solution of nickel and ferrous chlorids. It is also desirable in using this electrolyte that the anode used should contain as small a percentage of sulfur as is practicable, to keep down the formation of 110 sulfates, and these, if formed, may be removed by the occasional addition to the solution of barium chlorid or equivalent reagent. Should cobalt be present with the nickel it will react in the same manner in each of the 115 processes herein set forth, and whenever nickel is referred to, it is to be understood to include nickel and cobalt in combination. Frequently the anode mud, formed of constituents insoluble in the electrolyte, exhib- 120 its sufficient cohesiveness to be removed with the nearly dissolved anode. It should not be permitted to remain intact on the anode beyond a certain thickness as the impurities may become an insoluble anode which would 125 disturb the electrolyte balance of the solution, as chlorin instead of attacking the metal constituents of the anode would appear as free chlorin and oxidize the salts in solution, which it has been pointed out 130

969,773

should be maintained in their reduced condition. Some of the mud may settle in the bottom of the anode compartment, and some may remain in suspension in the electrolyte from which it will be removed by the filters. Suitable filters, settling tanks, or heaters may be introduced in or between any of the

vats or receptacles.

It may be found necessary in carrying out 10 my process in some instances to introduce into the electrolytes a small amount of organic compounds, weakly dissociated acids, or salts, to improve the quality of the cathode deposit or to increase the conduc-15 tivity of the electrolyte; also to add small amounts of free acids to prevent the formation of basic salts or to redissolve them if formed. But in making these additions of acids care should be taken to so limit them 20 as to maintain the electrolyte in a substantially neutral condition. It is not necessary that the metals of the so-called alloys be present therein in an alloyed state, as they might be mixtures, and when I refer to 25 alloys in the claims, I mean to include metals chemically or mechanically combined.

Any suitable form of cathode can be employed in the first vat A for receiving the deposit of nickel and iron, but I have dissovered that if the initial deposit is made upon a cathode so constituted as to hold it intact mechanically, for example, a cathode of wire mesh, gauze or netting, the deposit, which, if obtained by present methods shows a strong tendency to strip or peel off, can be carried to considerable thickness without stripping, as the stripping takes place between the deposit and the original cathode and not between the particles of layers of the

40 deposited metal.

What I claim as new, and desire to secure by Letters Patent of the United States, is—

1. The process of eliminating copper from alloys containing nickel and copper which consists in bringing the original alloy into solution electrolytically, substituting iron for copper in this solution, and electrolytically depositing nickel and iron from the solution thus obtained, substantially as described.

2. The process of climinating copper from alloys containing nickel and copper which consists in bringing the original alloy into solution electrolytically, substituting nickel and iron for copper in this solution, and electrolytically depositing nickel and iron from the solution thus obtained, substan-

tially as described.

3. The process of eliminating copper from alloys containing nickel and copper which consists in bringing the original alloy into solution electrolytically so that the copper is in a cuprous state and any iron present is in a ferrous state, substituting iron for copper in this solution and electrolytically de-

positing nickel and iron from the solution thus obtained, substantially as described.

4. The process of eliminating copper from alloys containing nickel and copper, which consists in bringing the original alloy into 70 solution electrolytically, so that the copper is in a cuprous state, substituting iron for copper in this solution so that the iron salts are in a ferrous state, and electrolytically depositing nickel and iron from the solution 75 thus obtained, substantially as described.

5. The process of eliminating copper from alloys containing nickel and copper which consists in bringing the original alloy into solution electrolytically so that the copper is 80 in a cuprous state and any iron present is in a ferrous state, substituting nickel and iron for copper in this solution, and electrolytically depositing nickel and iron from the solution thus obtained, substantially as 85 described.

6. The process of eliminating copper from alloys containing nickel and copper which consists in bringing the original alloy into solution electrolytically, substituting iron 90 for copper in this solution by cementation, and electrolytically depositing nickel and iron from the solution thus obtained, sub-

stantially as described.

7. The process of eliminating copper from 95 alloys containing nickel and copper which consists in bringing the original alloy into solution electrolytically, substituting iron for copper in this solution by selective deposition and cementation, and electrolytically depositing nickel and iron from the solution thus obtained, substantially as described.

8. The process of eliminating copper from alloys containing nickel and copper which 105 consists in bringing the original alloy into solution electrolyically, substituting nickel and iron for copper in this solution by selective deposition and cementation, and electrolytically depositing nickel and iron from 110 the solution thus obtained, substantially as described.

9. The process of climinating copper from alloys containing nickel and copper, which consists in connecting the alloy as the anode 115 in an electrolyte comprising a concentrated substantially neutral aqueous solution of nickel and ferrous chlorids, withdrawing the solution thus formed, substituting iron for copper so that the iron shall be present 120 in a ferrous condit on, and electrolytically depositing nickel and iron from the solution thus obtained, substantially as described.

Signed by me at New York, N. Y., this 10th day of November, 1909.

## · PERCY FOOTE COWING.

Witnesses:

FRANK C. COLE, Samuel W. Balch.