

UNITED STATES PATENT OFFICE.

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ART OF SEPARATING COPPER FROM OTHER METALS.

1,050,629.

Specification of Letters Patent. Patented Jan. 14, 1913.

No Drawing.

Application filed May 9, 1911. Serial No. 626,298.

To all whom it may concern:

Be it known that I, THOMAS A. EDISON, a citizen of the United States, and a resident of Llewellyn Park, West Orange, in the county of Essex and State of New Jersey, have invented certain new and useful improvements in the Art of Separating Copper from other Metals, of which the following is a specification.

10 In Letters Patent of the United States No. 936,525, dated October '12, 1909, and Nos. 865,687 and 865,688, dated September 10, 1907, I have described processes for making metallic films or flakes, and particularly
15 films or flakes of metallic nickel for use in the make-up of the positive electrodes of storage batteries of the Edison type. These processes consist generally in forming composite sheets composed of alternating layers
20 of electrolytically deposited copper and nickel, cutting up the sheets into strips or other small portions, and dissolving out the copper. In the hereinbefore mentioned Patent No. 865,687, I have described the use
25 of an ammoniacal copper sulfate solution for dissolving the copper without affecting the nickel.

My present invention relates generally to an improved process for separating copper
30 from another metal or metals associated therewith, by dissolving out the copper and leaving the other metal or metals unaffected.

More specifically, my invention relates to an improved process for separating copper
35 and nickel which are associated together in their metallic states, by dissolving out the copper and leaving the nickel unaffected.

My improved process is particularly adapted to the manufacture of films or flakes
40 of metallic nickel for use in the make-up of the positive electrodes of storage batteries of the Edison type.

I have discovered that metallic copper is readily acted upon by a strong solution of
45 sulfate of ammonia, containing a small quantity of a reducible chlorid such as cupric chlorid; that the rapidity of the action is increased very considerably by stirring and heating; and that the reducible
50 chlorid may be regenerated by supplying the solution with oxygen-containing gas, such as air or oxygen, and the operation thereby rendered continuous without the necessity of adding additional quantities of reducible
55 chlorid.

In the practice of my invention as applied

to the manufacture of films or flakes of metallic nickel, I proceed substantially as follows:—Composite sheets each composed of a thin layer of copper and a thin layer of
60 nickel or a number of such layers with copper and nickel alternating, are prepared by any of the methods described in the hereinbefore mentioned patents, or in any other
65 suitable manner. Each of these sheets is then cut into strips or into small portions corresponding to the size of the flake desired in the manner described in any of the hereinbefore mentioned patents. These strips
70 or other small portions, each consisting of copper and nickel associated together in their metallic states, are then put into a strong solution of sulfate of ammonia containing a small quantity of a reducible
75 chlorid such as cupric chlorid. I have found that excellent results are obtained by using a solution containing 285 grams of ammonium sulfate and 10 grams of cupric chlorid per liter. These proportions may be varied
80 within certain limits. The mixture containing this solution and the strips or other small portions of composite metal is then stirred and heated, and at the same time air is blown into it from any suitable source,
85 such as an air compressor. The heating of the solution may be accomplished by heating the air before it is forced in, or in any other suitable manner. The stirring may be
90 accomplished by suitable stirring apparatus, such as rotating vanes, or by the air which is forced in, or by both. As a result of the reaction, the copper is separated out as a basic sulfate of copper which is insoluble in the solution used, and which is held in suspension during the stirring. The nickel
95 flake collects at the bottom of the solution, and the solution together with the suspended basic sulfate may be poured or siphoned off. The basic sulfate is then separated from the solution by subsidence or filtration.
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In the reaction which takes place, the cupric chlorid apparently acts merely as a transferring agent for conveying an oxidizing element to the copper, first forming a monochlorid of copper, which is soluble in
105 ammonium sulfate. The air which is blown into the mixture serves to regenerate the cupric chlorid.

Ferric chlorid may be employed instead of cupric chlorid, but the results are less
110 satisfactory. Other reducible haloids may be used to take the place of the cupric or

ferric chlorid, such as cupric bromid, and ferric bromid, but these salts are more costly than the chlorids.

In the process employed heretofore in which the copper is dissolved in an ammoniacal solution of copper sulfate, it is necessary to use a large quantity of free ammonia. The ammonia is expensive and even though it may be recovered afterward from the solution, the process of recovering it requires complicated apparatus and is expensive. Furthermore, the operation of dissolving copper in the ammoniacal solution must be carried on where air can reach the solution to insure the dissolving of the copper. As a result, the solution cannot be heated to hasten the reaction, because, if heated too much ammonia would be driven off. Even when carried on without heating, fumes of ammonia are given off which fill the building and produce disagreeable and irritating effects. In my improved process, no free ammonia is used, and there are no fumes of ammonia perceptible. In fact, the solution is slightly acid. Furthermore, in the process employed heretofore, the copper is converted into a soluble salt and its recovery in a useful form is expensive. In my improved process the copper is automatically thrown out as an insoluble compound, that is, as a basic copper sulfate, and the solution can be used continuously by filtering off the basic salt and adding a quantity of sulfate of ammonia corresponding to the amount of sulfate removed in the basic salt. As the cupric chlorid is regenerated during the reaction, and remains in the liquid, it is only necessary to add a little from time to time to replace what is lost in the operation of filtering.

The copper may be recovered in a useful form from the basic copper sulfate by boiling the latter in a solution of sodium hydroxid or other alkali, whereupon the copper will be thrown down as the black oxid of copper.

My improved process is applicable generally to the separation of copper from other metals which are not affected by the treatment hereinbefore described.

Having now described my invention, what I claim as new and desire to protect by Letters Patent is as follows:—

1. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a reducible haloid of a metal of variable valence, substantially as described.

2. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a reducible

haloid of a metal of variable valence, and heating the mixture, substantially as described.

3. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a reducible haloid of a metal of variable valence, and stirring the mixture, substantially as described.

4. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a reducible haloid of a metal of variable valence, and heating and stirring the mixture, substantially as described.

5. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a reducible haloid of a metal of variable valence, and supplying oxygen containing gas to the mixture, substantially as described.

6. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a reducible haloid of a metal of variable valence, and supplying heated oxygen-containing gas to the mixture, substantially as described.

7. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a reducible haloid of a metal of variable valence, and simultaneously stirring, heating, and supplying oxygen-containing gas to the mixture, substantially as described.

8. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a cupric haloid, substantially as described.

9. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a cupric haloid, and heating the mixture, substantially as described.

10. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a cupric haloid, and stirring the mixture, substantially as described.

11. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associ-

ated metals with a solution of ammonium sulfate containing a cupric haloid, and heating and stirring the mixture, substantially as described.

5 12. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a cupric haloid, and supplying oxygen-containing gas to the mixture, substantially as described.

10 13. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a cupric haloid, and supplying heated oxygen-containing gas to the mixture, substantially as described.

15 14. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing a cupric haloid, and simultaneously stirring, heating, and supplying oxygen-containing gas to the mixture, substantially as described.

20 15. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, substantially as described.

25 16. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, and heating the mixture, substantially as described.

30 17. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, and stirring the mixture, substantially as described.

35 18. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, and heating and stirring the mixture, substantially as described.

40 19. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, and supplying oxygen-containing gas to the mixture, substantially as described.

45 20. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, and supplying heated oxygen-containing gas to the mixture, substantially as described.

21. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, and simultaneously stirring, heating, and supplying oxygen-containing gas to the mixture, substantially as described. 70

22. In a process of separating metallic copper from another metal or metals associated therewith, the conversion of the copper into a basic sulfate of copper, and the subsequent heating of the basic sulfate of copper in a solution of alkali, whereby black oxid of copper is formed, substantially as described. 75

23. The process of separating metallic copper from metallic nickel associated therewith, which consists in treating the associated metals with a solution of ammonium sulfate containing cupric chlorid, stirring, heating, and supplying air to the mixture, whereby the copper is converted into an insoluble salt, and then removing the said salt, substantially as described. 80

24. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution containing a reducible haloid of a metal of variable valence, said solution being capable of reacting with copper in the presence of oxygen to first dissolve the same and then to form an insoluble compound thereof, and supplying oxygen-containing gas to the mixture, substantially as described. 85

25. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution containing a reducible haloid of a metal of variable valence, said solution being capable of reacting with copper in the presence of oxygen to first dissolve the same and then to form an insoluble compound thereof, heating the mixture, and supplying oxygen-containing gas thereto, substantially as described. 90

26. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution containing a reducible haloid of a metal of variable valence, said solution being capable of reacting with copper in the presence of oxygen to first dissolve the same and then to form an insoluble compound thereof, and stirring, heating, and supplying oxygen-containing gas to the mixture, substantially as described. 100

27. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of a suitable sulfate containing a reducible 105

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haloid of a metal of variable valence, said solution being capable of reacting with copper in the presence of oxygen to form an insoluble compound of copper, and supplying oxygen-containing gas to the mixture, substantially as described.

28. The process of separating metallic copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of a suitable sulfate containing a reducible haloid of a metal of variable valence, said solution being capable of reacting with copper in the presence of oxygen to form an insoluble compound of copper, heating the mixture, and supplying oxygen-containing gas thereto, substantially as described.

29. The process of separating metallic

copper from another metal or metals associated therewith, which consists in treating the associated metals with a solution of a suitable sulfate containing a reducible haloid of a metal of variable valence, said solution being capable of reacting with copper in the presence of oxygen to form an insoluble compound of copper, and stirring, heating, and supplying oxygen-containing gas to the mixture, substantially as described.

This specification signed and witnessed this 1st day of May, 1911.

THOMAS A. EDISON.

Witnesses:

HENRY LANAHAN,
ANNA R. KLEHM.