March 1, 1949. A. E. LUNDBYE 2,463,190

METHOD OF PRODUCING NICKEL POWDER FROM PRINTING PRESS PLATES AND THE LIKE

Filed July 1, 1946

FIG. 1.

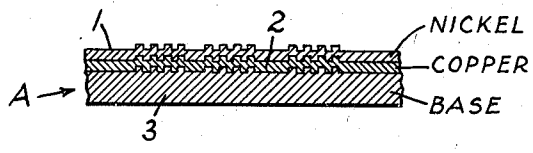


FIG. 2.



FIG. 3.

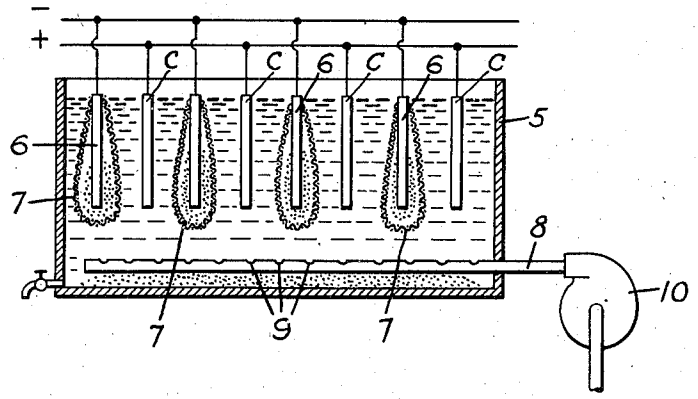


FIG. 4.

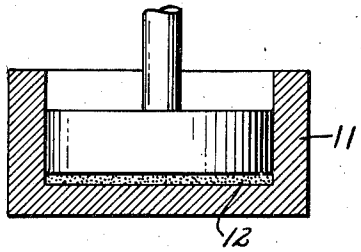
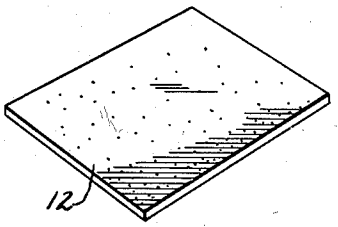


FIG. 5.



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Patented Mar. 1, 1949

2,463,190

UNITED STATES PATENT OFFICE

2,463,190

METHOD OF PRODUCING NICKEL POWDER FROM PRINTING PRESS PLATES AND THE LIKE

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Application July 1, 1946, Serial No. 680,790

5 Claims. (Cl. 204—10)

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My invention relates to new and useful improvements in the method of producing nickel powder from reclaimed scrap in which nickel is one of the elements.

The present application is a continuation-in-part of application Serial No. 447,582, filed June 18, 1942, now abandoned, and a companion to an application filed by me on May 23, 1942, bearing Serial No. 444,228, and entitled "Method of and apparatus for the separation and reclamation of copper from printing plates and the like," now Patent No. 2,356,329, dated August 29, 1944, wherein the recovery of nickel was mentioned and wherein the invention was directed more specifically to the recovery of copper.

As is well known to those skilled in the art, the letter press plates used in the large printing establishments are made up of a base metal, which is an alloy of lead, tin and antimony, which in turn has been cast on a copper plate, which copper plate in turn has been electrolytically deposited on a layer of nickel.

Stating it in another manner, in making up these printing plates, the original copper etching is pressed against a mold of lead or wax which mold is then electroplated with a layer of nickel which then is electroplated with copper, after which the mold with its nickel and copper deposits is withdrawn from the tank and the mold stripped from the plates. The plates, now consisting of nickel and copper, are turned with the copper surface up and a layer of an alloy of lead, tin and antimony is cast thereon.

An object of the present invention is to provide a method of recovering substantially pure metallic nickel in powdered form from a plate or other article having layers of nickel and copper thereon.

Still another object of the invention is to provide a method wherein the nickel may be stripped from the plates or other nickel and copper-plated articles in a relatively short length of time, purified and deposited as a finely divided powder which is suitable for the production of nickel electrodes of sufficient purity to be used in nickel-plating operations.

With these and other objects in view, the invention consists in certain new and novel methods and steps in the process of recovery as will be hereinafter more fully described and pointed out in the claims.

In the drawings,

Figure 1 is a fragmentary section of a plate from which the nickel is to be recovered;

Figure 2 diagrammatically shows the steps in the recovery;

Figure 3 diagramatically shows the electrolytic tank when arranged for the deposition of powdered nickel on suitable cathodes;

Figure 4 diagrammatically illustrates the compressing of the powder into the form of an anode, and

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Figure 5 is the anode in its completed form.

It will be understood that only that much of a flow sheet or diagrammatic views are set out as is thought necessary to illustrate the practice of the invention.

As heretofore mentioned, printing plates A are generally made up of a nickel shell or plate 1 which was originally electrolytically deposited on a mold on which there has been formed an imprint by the original copper etching plate.

Electrolytically deposited on this nickel is the copper plated shell 2 while on the rear of this plate for tinning purposes and not shown in the present view is a layer of tin for bonding the copper shell to the base metal 3, which consists of electrotype metal made up of an alloy of lead, tin and antimony.

The plates A are sometimes in a curved form, rather than flat, as shown, but as pointed out in Patent No. 2,356,329 (the companion application) heretofore referred to, the plates may be flattened before being treated for the recovery of the nickel, copper, and the base metal—that is, the lead, tin and antimony.

As also mentioned in the Patent No. 2,356,329, before the electrolyte treatment of the plates is begun, they are thoroughly washed in a hydrocarbon, such as kerosene, to remove the ink and other impurities, and a tank 4 for this purpose is shown in Figure 2 of the drawings. The plates A may, of course, be brushed with a stiff metal wire brush after being dipped in the washing tanks. They should then be cleaned with a solvent, such as hot, soapy water.

After all the ink and impurities have been removed from the plates A, they are supported by hangers in a tank 5 which contains an electrolytic stripping solution, and the tank may be provided with stainless steel cathodes 6 which cooperate with the plates A that form the anodes. An economical size tank is one that will hold 450 gallons and can be successfully operated by two men. A tank of the size mentioned will hold about twenty-eight plates.

The stripping solution may consist of a 10% to 25% solution of muriatic acid, HCl, preferably about a 10% solution, containing about 3 ounces of boric acid per gallon of solution inasmuch as this bath removes the nickel from the plates quicker than other baths containing HCl under similar conditions of temperature and current density. Good results are obtained, however, with stripping baths containing as little as 3% hydrochloric acid or as much as 25% hydrochloric acid. Also, the quantity of boric acid can be varied considerably; for example, between about 2 ounces per gallon and 5 ounces per gallon.

The boric acid acts as a buffer medium to

control the pH value of the solution. Other suitable buffer mediums such as citric acid, acetic acid and sulfuric acid may be used, but I have obtained very satisfactory results with boric acid.

The electric current may be of three to four volts with a maximum of 400 amperes, and I have found that under ordinary conditions the nickel may be stripped from the plates A in four minutes or less. Under ideal conditions the nickel can be stripped from the plates in about fifteen seconds.

After the nickel has been stripped from the plates, they are washed under running water to remove all traces of the solution.

The corners of the plates, which were not subjected to the stripping action, may be sheared off and the nickel also removed from these shearings.

The remaining plate, that is, the copper with its backing of lead, antimony and tin, is then subjected to the process set forth in the Patent No. 2,356,329 to recover the copper and form the same into anodes and to also recover the base metal.

Referring again to the gist of the present invention, it will be understood that since the tank **5** is of about 450 gallon capacity, it can contain approximately 1000 pounds of nickel. Due to the great solubility of the nickel chloride, high concentrations of nickel may be built up in the stripping tank, for example as much as 36 ounces to the gallon.

Before the nickel is plated out of the solution, the solution or electrolyte in the tank **5** must be treated with a hydrogen sulfide gas, ammonium sulfide or any equivalent reagent, which precipitates all the copper, lead, tin and antimony as sulfides but leaves the nickel in the acid solution as nickel chloride. Ammonium sulfide may be introduced into the bath in a plurality of streams and mixed with the bath. The pH value of the bath should not exceed 4 in order to avoid precipitation of nickel sulfide.

If hydrogen sulfide is used, I provide a pipe **8** provided with the apertures **9** which connects with a blower **10** to bubble or force the hydrogen sulfide gas through the solution. This is necessary as in the stripping of the nickel from the plate there is a small amount of lead, copper, tin and antimony which will enter the solution and must be removed.

The stripping solution may be treated with this hydrogen sulfide gas or other sulfide reagent at least once every week, and the solution filtered thus removing as sulfides all undesirable ions such as copper, lead, tin and antimony.

The duration of the treatment of the solution and the quantity of sulfide reagent required to precipitate the impurities from the solution can be determined by removing a sample from the tank and filtering to obtain a clear solution. If hydrogen sulfide is then passed through or other suitable sulfide reagent is added to this clear, filtered sample and no further precipitation takes place, all the undesirable metallic ions have been precipitated. It must be made certain that the stripping solution is acid before the hydrogen sulfide is introduced. Loss of nickel as nickel sulfide is avoided by maintaining the bath of a pH value of 4 or lower.

After all the copper, lead, tin and antimony have been precipitated as sulfides, the solution should be heated to about 150° F. and filtered, after which the filtrate is boiled to drive off any excess hydrogen sulfide.

Powdered nickel is deposited from the bath in the following way. A plurality of carbon electrodes C are placed in the hangers previously used for supporting the plates A during the stripping operation and the stainless steel cathodes **6** are provided with pervious bags **7** which are not attacked by the bath. The bags **7** are used to catch any of the deposited powdered nickel that may drop from the electrodes **6**.

The nickel may be deposited at a suitable current density, depending upon the type of the deposit desired. Powdered nickel can be deposited at a current density of about 3 to 5 amperes per square inch and a temperature of about 68° to 75° F. Such a high current density is used to prevent the deposition of a bright nickel plate which is deposited at a current density of about .2 to .3 ampere per square inch.

After the nickel has been deposited on the cathodes **6**, they are removed from the tank **5**, the recovered nickel is scraped from the cathodes, removed from the bags, washed and dried and made ready for the next operation. The recovered nickel is in the form of a very finely divided black magnetic powder.

If the nickel powder is to be formed into anodes, the powder is placed in the chamber or barrel of a press **11** and is subjected to a pressure of from between eight to fifty tons per square inch, so that the nickel is now compressed into a solid anode **12**.

It will be understood that the shape or form of the anode **12** may be as desired.

Finally, it will be understood that though I specifically stress that the nickel is reclaimed from printing press plates, the nickel might be reclaimed from other metal objects of a similar nature wherein there is a quantity of nickel to be recovered.

From the foregoing it will be seen that I first procure or strip the nickel from printing plates or similar objects which have heretofore been discarded for scrap, and then recover the nickel in its powdered form and subject the same to a great pressure to form anodes that give superior results to the rolled nickel anodes now in general use.

Having thus described my invention, what I claim as new and desired to secure by Letters Patent is:

1. The method of forming nickel powder from printing plates, which consist of a layer of nickel, an underlying layer of copper and a base formed of an alloy of lead, antimony and tin underlying said copper layer, which consists in cleaning the plates; placing the plates in an electrolytic bath consisting of between about a 3% and 25% hydrochloric acid solution and containing between about 3 and 5 ounces of boric acid per gallon; electrolytically stripping the nickel from the plate as an anode; thereafter precipitating copper, tin, lead and antimony ions as sulfide compounds from said bath by introducing into said bath a fluid sulfide while maintaining the bath at a pH value lower than 4; separating the precipitated sulfide compounds from the acid electrolytic bath; and electrolytically depositing the nickel in powder form on a cathode from said acid electrolytic bath.

2. A method of forming nickel powder from printing plates which plates are made of a layer of nickel, an underlying layer of copper and a base formed of an alloy of lead, tin and antimony underlying said copper layer which consists in thoroughly cleansing the plates; placing the plates in an electrolytic bath consisting essen-

tially of a 3% to 25% hydrochloric acid solution containing a buffering agent; electrolytically stripping the nickel from the plates as anodes; passing hydrogen sulfide through the acid electrolytic bath to precipitate all the metals except the nickel while maintaining the pH value at a pH value not exceeding 4; heating and filtering said electrolytic bath to obtain an acid solution of nickel chloride; and electrolytically depositing nickel on a cathode from said acid solution at a current density between about 3 and 5 amperes per square inch to form nickel powder.

3. A method of forming nickel powder from printing plates which plates are made of a layer of nickel, an underlying layer of copper and a supporting base formed of an alloy of lead, tin and antimony underlying said copper layer which consists in thoroughly washing the plates, placing the plates in an acid electrolytic bath consisting of about a 10% solution of hydrochloric acid containing about 3 ounces of boric acid per gallon of solution, keeping the bath at about room temperature, passing a current of sufficient density through the bath to strip the nickel from the plate as an anode; passing hydrogen sulfide through the acid electrolytic bath to precipitate the copper and other ions; heating the acid electrolytic bath to approximately 150° F., filtering the same; and electrolytically depositing the nickel as a powder from said filtrate on a cathode at a current density between about 3 and 5 amperes per square inch.

4. The method of forming nickel powder from printing plates which plates are made up of a layer of nickel, an underlying layer of copper, and a supporting base formed of an alloy of lead, antimony and tin underlying said copper layer, which consists in thoroughly cleansing the plates, placing the plates as anodes in an electrolytic bath consisting of about 10% hydrochloric acid containing about 3 to 5 ounces of boric acid per gallon of acid solution, passing a current through the bath of sufficient density to strip the nickel from the plates, together with a small amount of other metallic ions; removing the plates; passing hydrogen sulfide through the acid bath to precipitate sulfides of the metals other than nickel

while maintaining the latter at a pH value of not more than about 4; heating the bath to approximately 150° F., filtering the same; and passing a current at a density between about 3 and 5 amperes per square inch therethrough to deposit the nickel in powder form on the cathode.

5. A method of forming nickel powder from articles having a layer of nickel superimposed on a layer of copper comprising immersing said articles in an electrolytic bath consisting of about a 3% to 25% solution of hydrochloric acid containing a buffering agent, electrolytically stripping said nickel layer and some of said copper from said article, removing said article from said bath, precipitating the stripped copper from said bath as copper sulfide at a pH value not exceeding 4 to avoid formation of nickel sulfide by introducing into said bath a fluid sulfide, and depositing nickel from said acid solution on a cathode at a current density between about 3 and 5 amperes per square inch to deposit the nickel as a powder.

AXEL E. LUNDBYE.

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