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ELECTRODEPOSITION OF METALS

Original Filed April 26, 1919

FIG. 1.

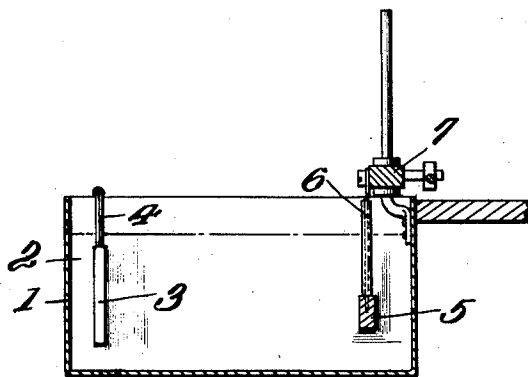
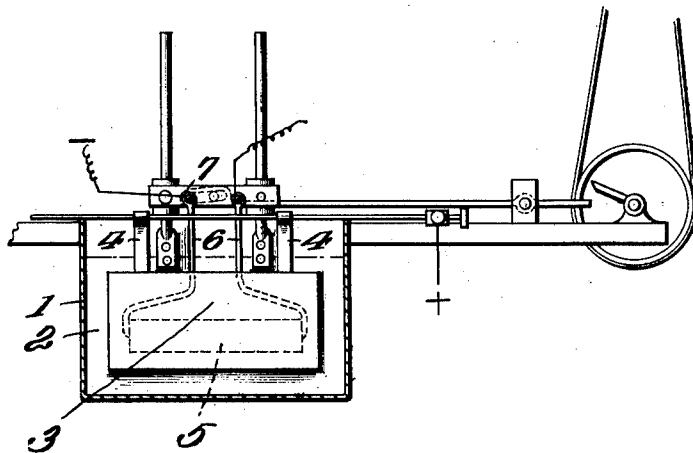


FIG. 2.

Witness

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UNITED STATES PATENT OFFICE.

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ELECTRODEPOSITION OF METALS.

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My invention relates to a method and means of electro-depositing metals, particularly the "nickel metals", by which I mean nickel, or cobalt, or alloys containing either nickel or cobalt or both of these metals. My invention aims to produce a new bath and a new method of employing said bath, whereby I am enabled to electrodeposit said "nickel metals" at a very high rate, using very high current densities. By means of my invention I am further enabled to substantially avoid "pitting" or "pits" in the deposits. By the use of my invention, moreover, said "nickel metals" may be obtained in a dense, reguline, homogeneous, malleable and ductile condition. Said "nickel metals" deposited in accordance with my invention, are substantially free from pits, pores and hydrogen, and are workable to a remarkable degree.

By way of example, I shall describe an illustrative embodiment of my invention in the following specification. I shall set forth my invention as applied more particularly to the electrodeposition of my new nickel, as described in my copending application Serial No. 292794, filed April 6, 1919, though it is of course to be understood that analogous means may be applied to the electrodeposition of the other "nickel metals."

In producing the foregoing new nickel I may use an electrodeposition bath of approximately the following composition: Nickel sulphate, 240 g.; nickel chloride, 20 g.; boric acid, 40 g.; water, 1 liter. This bath is preferably operated at a temperature of about 130° F. Except for the addition of boric acid, as set forth above, the bath should be neutral. The anode may consist of cast or rolled nickel, and the cathode of a metal, such as aluminum or nickel, which is adapted to receive a smooth, separable deposit of the electrodeposited nickel. However, if so desired, the metal may be electrodeposited adherently, as in the form of a plating.

During deposition the cathode on which the nickel is being electrodeposited, is intermittently or periodically exposed to a gaseous medium, such as the air, as by removal from the bath or otherwise. The time during which the cathode and the electrodeposited nickel carried by it are kept out of the bath should be more than what I term

the "minimum" or "hydrogen dissipation" period, which is necessary to permit the hydrogen deposited with the metal to be dissipated or removed by contact with the surrounding gaseous medium, such as the air. This "minimum" or "hydrogen dissipation" period may be readily determined by trial and experiment and is generally greater than about one second, usually two seconds.

The time during which the cathode and the deposit carried by it are kept out of the bath should not, however, exceed what I term the "maximum" or "critical" or "separable deposit" period, which, in the case here given, is from about 6 to about 16 seconds, by which I mean that if the time during which the cathode and its deposit are kept out of the bath exceeds the said "maximum" or "critical" or "separable deposit" period, on restoring the cathode and its deposit to the bath, the succeeding deposit will not be adherent but will separate or be easily separable from the previous deposit. This results in a laminated, weak metal, a condition which is obviously to be avoided, where a sound unitary metal is desired.

The frequency of removal or "exposure frequency period" is determined by the factors of deposition which influence hydrogen liberation, such as the degree of exactness of neutrality, current density, and temperature. I have found that in the bath here described by way of example, and operating at a temperature of about 130° F., and with a current density of about 10 amperes per square decimeter, this period may be from 1 to 2 minutes.

In order, moreover, that the electrodeposited metal such as the nickel in the example given, may not only be dense, reguline and free from pores and hydrogen, but in order also that said metal shall be free from what are known as "pits" in the art of the deposition of the "nickel metals", I add to the bath, preferably before starting the electrodeposition, a quantity of finely divided nickel hydrate, preferably freshly precipitated nickel hydrate (NiO_2H_2) and probably existing in a colloidal condition, which nickel hydrate remains in suspension in the bath during electrodeposition. While the quantity of hydrate so added may vary within considerable limits, I prefer to add from 1 to 5 grams, generally 1 gram, of said

colloidal nickel hydrate to each liter of bath solution.

I have discovered that by adding to the bath certain salts, such as the sulphates, of the alkali metals, preferably sodium, finely divided or colloidal nickel hydrate is produced and held in suspension in the bath. This freshly and continuously produced hydrate replenishes the hydrate which during the operation of the bath aggregates or for other reasons goes out of suspension and drops to the bottom of the bath. Such salts have further beneficial effects and greatly improve the operation of the bath and the character of the deposits formed. For this purpose I may add varying amounts, such as from about 1 to about 10 grams, preferably 1 gram, of sodium sulphate for each liter of bath solution.

The action of the colloid-producing substance, such as the sodium sulphate, described above, is to produce by and during electrodeposition, some caustic soda or sodium hydroxide. The caustic soda or sodium hydroxide thus produced, by interaction with the nickel salts present in the bath, such as the nickel sulphate, generates finely divided or colloidal nickel hydrate which remains suspended in the bath. The interaction at the same time re-generates the sodium sulphate. The sodium sulphate or its equivalent also acts to assist the deposition and to improve the character of the deposit, which factors are also further assisted by the initial addition of nickel hydrate in finely divided or colloidal condition to the bath.

In the accompanying drawing, I have diagrammatically illustrated a form of apparatus in which the foregoing illustrative embodiment of my invention may be carried out.

Referring to these drawings:

Figure 1 is a cross section; and

Figure 2 is a longitudinal section of such apparatus.

The apparatus comprises briefly, a vat 1, for containing the bath 2. 3 is the anode and 4 the connector for supplying said anode with current. The cathode is indicated at 5 and its conductor at 6. At 7 is indicated generally any suitable means, which may, if desired be manually operated, for intermittently or periodically removing the cathode from the bath, as set forth above.

The resulting product, as in the case of nickel in the illustrative embodiment set forth above, is, as before stated, dense, regular, homogeneous, malleable and ductile. The new nickel herein described is substantially free from pores and pits and contains no appreciable amount of hydrogen.

It is, of course, to be understood that where a deposit of cobalt, having the foregoing desirable properties, is desired to be produced, a cobalt anode is used in place of the nickel

anode, and cobalt compounds are used in place of the corresponding nickel compounds. Where alloys of either nickel or of cobalt or of both of these metals are desired to be produced, means analogous to the means herein set forth for the deposition of nickel and of cobalt in the desired condition and having the desired properties may be employed.

The nickel herein described differs in character from the nickel obtained by the usual metallurgical processes in that my new nickel is free from the usual "poisonous" contents such as sulphur, silicon, arsenic, carbon, carbides, oxygen, and other gases, and oxide containing compounds, generally present in metallurgical nickel and rendering the same imperfect and more difficult to work.

Furthermore, my new nickel is to be further differentiated from the ordinary metallurgical nickel in that it has a finer and more even grain than such metallurgical nickel, as is shown by metallographic micro-photographs. It is also considerably purer than such metallurgical nickel, resists chemical action better and has a higher melting point.

The nickel described herein is also to be differentiated from the usual electrodeposited nickel in that it is substantially free from pores, pits and hydrogen, and is malleable and ductile to a very high degree, unlike the usual electrodeposited nickel which is porous, contains a large hydrogen content and is brittle and unworkable.

A further difference in character between my new nickel and the ordinary metallurgical nickel is shown by the fact that the film of oxide formed by heating my new nickel in an oxidizing atmosphere is thinner, finer in texture and more adherent and flexible than the oxide coating formed by similar treatment of ordinary metallurgical nickel. Furthermore, such oxide coating in the case of my new nickel is of a different color, generally yellow to brown, while the oxide coating on the metallurgical nickel is generally violet or indigo in color.

It is of course, to be understood that the invention is not to be limited to the specific illustrative embodiment herein described for purposes of example only. It is also to be understood that the applicant does not wish to have the invention or the appended claims in any way limited by any particular theory of operation which he may now hold and which may be suggested by the foregoing detailed description.

What I claim is:

1. In the electrodeposition of "nickel metals", a bath comprising a salt of a "nickel metal" in solution, and a finely divided hydrate of a "nickel metal" in suspension therein.

2. In the electrodeposition of nickel, a bath comprising a salt of nickel in solution,

and a finely divided hydrate of nickel in suspension therein.

3. In the electrodeposition of "nickel metals", a bath comprising, in solution, a salt of a "nickel metal", and an agent adapted during electrodeposition to produce a finely divided hydrate of a "nickel metal" in suspension in the bath.

4. In the electrodeposition of nickel, a bath comprising, in solution, a salt of nickel, and an agent adapted during electrodeposition to produce a finely divided hydrate of nickel in suspension in the bath.

5. In the electrodeposition of nickel, a bath comprising, in suspension, finely divided nickel hydrate, and, in solution, a salt of nickel, and an agent adapted during electrodeposition to produce a finely divided hydrate of nickel in suspension in the bath.

6. A method of electrodepositing "nickel metals" which comprises intermittently elec-

trodepositing a "nickel metal" on the cathode from a bath containing, in solution, a chloride of a "nickel metal" an agent adapted during deposition to provide a finely divided hydrate of a "nickel metal" in suspension in said bath, and boric acid, and, in suspension, a finely divided hydrate of a "nickel metal", and successively bringing the surfaces of the successive deposits into contact with a gaseous medium for a period of time sufficiently long to permit the hydrogen in the deposited surfaces to be dissipated, but not long enough to cause the successive deposits to be separable, whereby a dense, homogeneous, unitary "nickel metal" deposit is formed.

In testimony whereof, I have signed my name to this specification this 26th day of April, 1919.

CHARLES P. MADSEN.