GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17.

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Index at acceptance—70C4 [LVIII(5)].

PROVISIONAL SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO NICKEL ELECTRODEPOSITION.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated under the Registration of Societies Act (Act XXI of 1860).

The following specification describes the nature of this invention.

This is an invention by Balkunje Anantha Shenoi, Scientist, Ramachandra Subramanian, Senior Scientific Assistant, both of the Central Electrochemical Research Institute, Karaikudi, Chelliah Balasingh, Senior Scientific Assistant, National Aeronautical Laboratory, Bangalore, and Venkataraman Srinivasan, Senior Scientific Assistant, National Chemical Laboratory, Poona, India, all Indian citizens.

This invention relates to improvements in or relating to the nickel plating from a Watt's type of nickel bath.

Hitherto it has been proposed to use low current densities in such a bath for plating and electro-forming applications.

This is open to the objection that the process is time consuming taking nearly one hour for plating a thickness of .001".

The object of this invention is to obviate these disadvantages by using very high current densities of the order of 300 as for more by use of modulated currents.

To these ends, the invention broadly consists in using a suitable combination of alternating current and direct current with a proper voltage ratio in a simple Watt's type of nickel plating baths. The d.c./a.c., current ratio can be varied from 7:1 to 1:1 and time cycle can be varied 5:1 to 1:1.

The following typical examples are given to illustrate the invention:

EXAMPLE 1.

The plating bath consists of nickel sulphate—300 g/l, nickel chloride—52 g/l. and boric acid—30 g/l, pH 3.5, Temperature 35-50° C. Nickel anodes are used. Filtered air agitation. D.C. to A.C. current ratio 7:6. The time cycle ratio is 4:1. A.C. is fed from mains 230V/50c/s, through a dimmerstat. D.C. is fed from a rectifier. The cathode is made of mild steel or copper or brass.

The following are among the main advantages of the invention:

1. A thick deposit of .004" could be obtained in a matter of 20 minutes.
2. The deposit is free from nodules, surface roughness and does not crack when flexed or bent.
3. The deposit does not become dark or darkmat even at high current densities.
4. Can be adapted to existing plating installations very easily.
5. Saves a lot of time and hence increase the working capacity of tank, nearly 5 to 6 times.

R. BHASKAR PAI.

Patents Officer,

Council of Scientific and Industrial Research.

Dated this 21st day of September 1965.

COMPLETE SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO NICKEL ELECTRODEPOSITION.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated under the Registration of Societies Act (Act XXI of 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This is an invention by Balkunje Anantha Shenoi, Scientist, Ramachandra Subramanian, Senior Scientific Assistant, both of the Central Electrochemical Research Institute, Karaikudi, Chelliah Balasingh, Senior Scientific Assistant, National Aeronautical Laboratory, Bangalore, and Venkataraman Srinivasan, Senior Scientific Assistant, National Chemical Laboratory, Poona, India, all Indian citizens.

This invention relates to improvements in or relating to the nickel electrodeposition from the Watts type of nickel baths.

Hitherto it has been proposed to use current densities in the range of 2 to 15 asdm in the Watts type of nickel plating baths and in the range of 2.5 to 30 asdm in the special types of baths like all chloride, sulphamate or fluoborate for electroplating and electroforming applications.

This is open to the following objections:

(a) The use of current density in the upper limits in the Watt's type of bath leads to unsatisfactory deposits causing pitting, burning and brittleness in the electrodeposited nickel, when the direct current alone is used.

(b) The special types of baths are either very costly or required special equipments for tank linings, heating coils and cooling coils because they are highly corrosive.

(c) The time required to build up satisfactory deposits of heavy nickel is long generally the rate of build up being about 0.001" in the current density range of 4 asdm.

(d) The use of current densities in the upper limits of the current density range generally resulted in an imbalancing of the bath due to improper anode dissolution.

The main object of this invention is to obviate these disadvantages caused by high current density electrodeposition in the conventional D.C. method by use of modulated currents during electrodeposition from the conventional Watts type of nickel baths. This permits to electrodeposit nickel from the Watts type of baths over a wide range of high current densities of 20 to 50 asdm without causing burning, pitting or brittleness. Thus the use of special type of baths like sulphamate, fluoborate or high chlorides need not be resorted to increase the rate of build up.

To these ends, the invention broadly consists in using a suitable combination of alternating current and direct current with a proper voltage ratio for the electrodeposition of nickel on the cathode surface AC/DC from the Watts type of nickel plating baths to obtain a DC/AC current ratio in the limits 1:1 to 1:1. D.C. time cycles for plating can be varied over 5:1 to

1:1. Thus by proper choice of the parameters, *viz.*, the DC/AC voltage ratio, DC/AC current ratio and DC/AC time cycle ratio nickel can be electrodeposited at current densities of 30 asdm or more, from a nickel plating bath containing nickel sulphate, nickel chloride and boric acid. The anodes used are conventional rolled or electrolytic nickel anodes. The anode dissolution is quite uniform with no passivation of the surface, which is a common phenomena observed in the conventional simple DC method.

The rate of deposition is thus increased to 0.004" in 20 minutes of passing direct current by employing a combination of AC/DC in the said range; whereas by the conventional simple DC method only 0.001" thick deposit could be obtained in 60 minutes with a current density of 2 asdm. The deposits obtained by the use of a combination of DC/AC as described above are smooth and adherent without pits on the surface even when the thickness of the deposit is of the order of 0.008" or more.

Thus the other objects of the invention are:

(a) The technique can be used in building up of heavy nickel deposits for salvage of worn out parts, mis-machined parts and for surfacing of machine parts with a thick hard layer of nickel. This could be achieved in a shorter time than the conventional methods, resulting in an increased production rate.

(b) The technique can be used in nickel electroforming applications also, to build up thick nickel electroforms in much a short time without sacrificing the desired mechanical properties of the nickel electrodeposit. Such a high rate of deposition of nickel from the Watts type cannot be achieved by the conventional simple DC methods.

Thus the present invention consists of a method for electrodeposition of nickel from the Watts type of nickel electroplating baths at such high current densities as 30 asdm or more by using a suitable combination of AC and DC. The following is the detailed description of the process and the procedure by which it is to be carried out.

The constituents of the electrolyte for nickel deposition can be varied over the following ranges:

Nickel sulphate . .	120—400 gms/litre
Nickel chloride . .	30— 60 gms/litre
Boric acid . . .	25—50 gms/litre

and the operating conditions are:

pH (electrometric) . .	2—4.5
Temperature . .	45—65° C.
Agitation . . .	Filtered air agitation
Anodes . . .	Bagged nickel anodes

Rolled or electrolytic nickel is connected to the positive terminal of the DC source (a rectifier or a motor-generator) and the work piece is connected to the negative terminal of the same source.

The nickel anode terminal and the work piece terminal are now connected through a variable transformer to the terminals of AC mains. AC is fed from 230V/50C mains.

The AC/DC voltage ratio is so maintained as to keep the DC/AC current ratio in the optimum value. The DC/AC current ratio can be varied over the limits 7:1 to 1:1.

The DC/AC time cycle ratio can be varied over the limits 5:1 to 1:1.

The DC current density used for electrodeposition can be varied over the limits 2 asdm to 50 asdm.

The total time of depositions are adjusted to give the desired thicknes of the nickel deposits depending on the current density used and when the desired thickness is obtained both the AC and DC circuits are cut off and the work piece removed, rinsed and dried as usual.

The following typical examples are given to illustrate the invention:

Example.

The plating bath consists of nickel sulphate—300 gms/litre, nickel chloride—52 gms/litre and boric acid—30 gms/litre, pH 3.5 Temperature 35—50° C. Nickel anodes are used. Filtered air agitation. DC to AC current ratio 7:6. The time cycle ratio is 4:1. A.C. is fed from mains 230V/50C/s, through a dimmerstat/. DC is fed from a rectifier. The cathode is made of mild steel or copper or brass. Rate of build up: 0.008" in 30 ft of total plating time.

The following are among the main advantages of the invention:

1. A thick deposit of 0.004" could be obtained in a matter of 20 minutes.
2. The deposit is free from nodules, surface roughness and does not crack when flex or bent.
3. The deposit does not become dark or darkmat even at high current densities.
4. Can be adapted to existing plating installations very easily.
5. Saves a lot of time and hence increase the working capacity of tank, nearly 5 to 6 times.

We claim:

1. A process for the electro deposition of nickel, *e.g.*, for electroforming or salvaging from the Watts type of plating baths wherein is used modulated current with a suitable combination of DC/AC, *viz.*, the DC/AC current ratio in the range of 7:1 to 1:1 and DC/AC time cycle ratio in the range of 5:1 to 1:1 whereby heavy nickel deposition is achieved at current densities in the range of 2 asdm, to 50 asdm.

2. A process as claimed in Claim 1 wherein the nickel electroplating bath contains the following constituents in the range.

Nickel sulphate . .	120—400 gms per litre
Nickel chloride . .	30— 60 gms per litre
Boric acid . . .	25— 50 gms per litre

and the operating conditions being:

pH (electrometric) .	2—4.5
Temperature . .	45—65° C.
Agitation . . .	Filtered air agitation
Anodes . . .	Bagged nickel anodes.

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Dated this 28th day of July 1966.

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