

PROVISIONAL SPECIFICATION.

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IMPROVEMENTS IN OR RELATING TO BRIGHT NICKEL PLATING.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJ 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 RAMACHANDRAN SUBRAMANIAN and BALKUNIE ANANTHA SHENOI, both of the Central Electro-Chemical Research Institute, Karaikudi-3, Madras, India both Indian citizens.

This invention relates to improvements in or relating to Bright Nickel Plating from Watt's type Nickel electroplating baths.

Hitherto it has been proposed to use imported proprietary brighteners at heavy cost.

This is open to the objection that the know-how has to be got at heavy cost and much foreign exchange has to be spent in utilising these imported bright nickel baths.

The object of this invention is to obviate these disadvantages by using commonly available organic chemicals in India as brighteners.

To these ends, the invention broadly consists in using a Nickel plating bath containing Nickel Sulphate, Nickel Chloride and Boric acid with brighteners from the group of some drugs or their derivatives and coumarin or its derivatives. In the conventional Nickel bath of Watt's type the brighteners are used in concentration ranging from .0001 M to .0015 M.

The following typical examples are given to illustrate the invention:

EXAMPLE 1.

The electroplating is carried out in the usual way on a steel basis metal. After giving an undercoat of copper, nickel from this plating bath is deposited and after rinsing chromium, from a conventional bath is flashed for 3 to 5 minutes.

NiSO <sub>4</sub> ·6H <sub>2</sub> O	300 g/l
NiCl <sub>2</sub> ·6H <sub>2</sub> O	52 g/l
H <sub>3</sub> BO <sub>3</sub>	40 g/l
pH	3.6
Temp.	40°C—55°C
Thiazole derivative	.0006M
Coumarin	.0005M
Cathode current	
Thiazole derivative 1	.0006M

EXAMPLE 2.

Same as above	
NiSO <sub>4</sub> ·6H <sub>2</sub> O	240 g/l
NiCl <sub>2</sub> ·6H <sub>2</sub> O	45 g/l
Boric acid	40 g/l
pH	4.5 to 5
Temperature	35°C—55°C
Thiazole derivative 1	.0004 M
Coumarin	.0005 M
Cathode current	
density	20—60 asf

The following are among the main advantages of the invention:

- (1) The brighteners are freely available in India for other purposes.
- (2) The adhesion of Nickel to steel is very good.
- (3) The tensile stress is comparable to simple Watts Nickel without stress reducing agents.
- (4) Saves a lot of foreign exchange.

R. BHASKAR PAI.,

Patents Officer.

Council of Scientific & Industrial Research.

Dated this 24th day of August 1964.

COMPLETE SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO BRIGHT NICKEL PLATING.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJ 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 RAMACHANDRA SUBRAMANIAN and BALKUNJE ANANTHA SHENOI, both of the Central Electro-chemical Research Institute, Karaikudi-3, Madras, India, both Indian citizens.

This invention relates to improvements in or relating to Bright Nickel Plating from Watt's type nickel electroplating baths.

Even though a number of organic compounds have been used in small quantities as addition agents to get minor bright nickel deposits from a nickel plating baths, the most of them are beset with following difficulties. The nickel deposit which may be bright is in a highly stressed condition developing cracks which result in poor adhesion and poor resistance to corrosion. The compounds may deteriorate in use resulting either decrease in brightness or sometimes the deteriorated products may themselves become detrimental to the deposition of metal leading to poor adhesion, missing of deposits in certain regions, peeling, flaking, etc. The bright nickel deposits obtained from nickel plating baths containing some class of brighteners do not take up the subsequent chromium plating for decorative purposes—either there is no deposition of chromium or deposition of

chromium is in patches. For good functioning of the bath continuous filtration through a bed of activated charcoal is adopted. This treatment removes the organic contaminants particularly grease, oil or resins that might have found their way into the bath either from air or tank linings or from decomposition products of the addition agents. Mostly the brighteners are also adsorbed by the activated charcoal resulting in complete depletion of the brightener from the bath necessitating continuous addition of the brightener to the bath. This makes maintenance and control of the bath difficult. For many of the brighteners recommended, either there are no analytical methods or the methods are tedious and time consuming for the estimation and control of the brighteners used. These difficulties have been overcome by proper choice of formulation with a combination of organic substances to be used in nickel baths to enhance the brightness, to reduce the stress, to increase the adhesion, to reduce pitting and other incumbent disadvantages in technically advanced countries like U.K., U.S.A., U.S.S.R. and Europe. Hence hitherto it has been proposed to use imported proprietary brighteners at a heavy cost, mainly because there is no indigenous

Price : TWO RUPEES.

process available for exploitation by the Indian industrialists.

This is open to the objection that the know-how of these foreign proprietary processes has to be got at a heavy cost and much foreign exchange has to be spent in utilisation of these imported bright nickel baths.

The object of this invention is mainly to obviate the last disadvantage by formulating a combination of organic substances that are freely available in this country for use as brighteners in the nickel plating bath containing nickel salts and boric acid.

The second object of this invention is to formulate the combination of such indigenously available organic substance to obtain bright nickel deposits by electrodeposition from Watt's type of nickel bath which are in no way inferior though not superior to the nickel deposits obtained by use of the foreign proprietary processes.

Another object of the invention is to make the process simple in the maintenance and control of the organic brightener made easily by analytical methods that can be adopted in any plating shop.

To these ends, the invention broadly consists in using a nickel plating bath containing nickel sulphate, nickel chloride and boric acid with brighteners from the group of sulpha drugs or their derivatives and coumarin or its derivatives.

Thus, nickel plating is done on metals like mild steel, copper or brass using a nickel plating bath of Watts type, the ingredients of which may be varied as follows.

Nickel sulphate	240—400 gms per litre
Nickel chloride	40—60 " " "
Boric acid	30—60 " " "
Sulpha drugs or their derivatives	.0002M—.001M
Coumarin or its derivatives	.0002M—.001M

The organic drug or its derivative acts as the primary brightener which is readily made soluble in the plating bath and can be used fairly over a wide concentration range without loss in the brightness of the deposit.

The coumarin or its derivatives belongs to the second class of brighteners used in conjunction with the primary brighteners in such concentration limits to obtain acceptable deposits of nickel without peeling or flaking.

A special feature of the above class of brighteners is that they are easily estimated by conventional colorimetric methods using abridged type of colorimeters even in an ordinary plating shop and thus control of brightener concentration is made easy. An added advantage of the said class of brighteners is that only negligible amount of brightener is lost during filtration through activated charcoal and hence the bath can be continuously filtered through a bed of activated carbon for removal of organic impurities. Since deposition of chromium for decorative purposes on the bright nickel deposited from the bath containing the said organic compounds properly formulated is adherent and coherent leaving no areas unplated, the use of the said bright nickel bath for decorative copper-nickel-chromium finishes on common basis metals like zinc or aluminium base die castings following the conventional methods of plating involving no special equipments is possible.

The following is the detailed description of the preparation of the brighteners. An aqueous or an alcoholic solution of the sulpha drugs or their derivatives is prepared by dissolving a known quantity of the organic substance or its alkali salt. (Solution I).

An alcoholic solution of coumarin or its derivative is prepared to contain a known amount of the substances in a specified volume of the solvent (Solution II).

Nickel plating bath is prepared and purified by conventional methods by dissolving nickel sulfate, nickel chloride and boric acid in water. To this purified solution, a known calculated volume of Solution I is added with continuous agitation of the bath, followed by a known specified volume of Solution II and the bath is allowed to mix well and is now ready for use.

The proportion of the ingredients may be varied within the following ranges in an aqueous solution:

Nickel sulphate crystals	240—400 g/l
Nickel chloride crystals	40—60 g/l
Boric acid	30—60 g/l
Sulpha drug derivative	.0002M—.001M
pH of the bath	3 to 5

The following examples are given to illustrate the invention;

#### EXAMPLE 1.

The electroplating is carried out in the usual way on a steel basis metal. After giving an undercoat of copper, nickel from this plating bath is deposited and after rinsing, chromium from a conventional bath is flashed for 3 to 5 minutes.

NiSO <sub>4</sub> .6H <sub>2</sub> O	300 g/l
NiCl <sub>2</sub> .6H <sub>2</sub> O	52 g/l
H <sub>3</sub> BO <sub>3</sub>	40 g/l
pH	3.6
Temp.	40°C—55°C
Sulpha drug derivative 1	.0006M
Coumarin	.0005M
Boric acid	30—60

#### EXAMPLE 2

Same as above

NiSO <sub>4</sub> .6H <sub>2</sub> O	240 g/l
NiCl <sub>2</sub> .6H <sub>2</sub> O	45 g/l
Boric acid	40 g/l
pH	4.5 to 5
Temperature	35°C—55°C
Sulpha drug derivative 1	.0004 M
Coumarin	.0002 M
Cathode current density	20—60 asf

The solutions can be operated with air agitation also.

The following are among the main advantages of the invention:

- (1) The brighteners are freely available in India for other purposes.
- (2) The adhesion of nickel to steel is very good.
- (3) The tensile stress is comparable to simple Watts nickel without stress reducing agents.
- (4) Saves a lot of foreign exchange.

We claim:

1. A process for bright nickel plating which consists in using a nickel plating bath containing nickel sulphate, nickel chloride and boric acid with brighteners from the group of sulpha drugs or their derivatives and coumarin or its derivatives.

2. A process as claimed in Claim 1 wherein nickel plating is done on metals like mild steel, copper or brass using a nickel plating bath of Watts type, the ingredients of which may be varied as follows:

Nickel sulphate	240—400 gms per litre
Nickel chloride	40—60 " " "
Boric acid	30—60 " " "
Sulpha drugs or their derivatives	.0002M—.001M
Coumarin or its derivatives	.0002M—.001M

3. Bright nickel plated articles whenever plated by a process substantially as hereinbefore described.

R. BHASKAR PAI,

Scientist,

Council of Scientific & Industrial Research,

Dated this 19th day of May 1963.