OVERNMENT OF INDIA : THE PATENT OFFICE : 214, ACHARYAJAGADISH BOSE ROAD, CALCUTTA-17

Complete Specification No. 138564 dated the 20th October, 1975. Application and Provisional specification No. 1958/Cal/74 dated the 31st August 1974

Acceptance of the complete specification advertised on the 21st February 1976

Index at acceptance—188—[XXXIII (9)]

International classification—(C23C $^{1}/_{10}$

IMPROVEMENTS IN OR RELATING TO THE IMMERSION DEPOSITION OF NICKEL ON MILD STEEL FOR SUBSEQUENT DEPOSITION WITH COPPER

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH

RAFI MARG. NEW DELHI-1, INDIA, AN INDIA 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, Ramachandran Subramanian, Scientist and Ramasubbu Venkatachalam Senior Scientific Assistant, all employed in the Central Electrochemical Research Institute, Karaikudi—623003 and all are Indian Nationals.

This invention relates to improvements in or relating to the immersion deposition of nickel on mild steel for subsequent deposition with copper.

Hitherto, it has been proposed to directly deposit copper on mild steel from a copper cyanide strike solution or direct nickel on mild steel surface from Watt's nickel solution for subsequent deposition with copper from acid copper bath.

This is open to the objection that copper cyanide salt used in the strike solution is costlier and is poisonous and toxic in nature. So, it requires a high hygienic atmosphere that can be achieved by the use of special fumehoods and fans. An initial deposition of copper from cyanide strike solutions or nickel from watt'snickel solutions is necessarily given on mild steel surface before regular thicker copper deposit from an acid copper bath. For this, extra busbar connections and rectifier are needed which add to the cost of plating.

The object of this invention is to obviate these disadvantages by using immersion nickel deposit on mild steel surface before regular copper deposition from acid copper bath, at a temperature of 70-80°C for a period of 10-75 minutes with stirring.

To these ends, the invention broadly consists in immersing the article to be plated in a solution of nickel chloride and boric acid containing an organic addition agent with one or more alcoholic groups or its polymerised product.

The following typical examples are given to illustrate the invention :

EXAMPLE- 1

1 Degrease the polished surface of mild steel

2 Cathodic electroclean in an alkali cleaner in the temperature range 60-70°C with current density of 144 asf .

3 Rinse

4 Immersion nickeling in the solution given below

$NiCl_2$. $6H_2O$	:	500 g/l
H_3BO_3	:	25 g/l
Glycerol	:	75 ml/l
pH	:	2.1-3.5 (Electrometric)
Temperature	:	75°C
Time	:	10 minutes

5 Rinse

6 Copper in conventional acid copper bath of the composition given below :

$CuSO_4$. $5H_2O$	:	200 g/l
H_2SO_4	:	75 gm/l
Glue	:	0.1 g/l

7 Rinse and dry

EXAMPLE 2

Except the sequence (4), the other operations are the same as in Example 1.

4	$NiCl_2$. $6H_2O$	:	700 g/l
	H_3BO_3	:	40 g/l
	Glycol	:	100 g/l
	pH	:	2.1-3.5 (Electrometric)
	Temperature	:	80°C
	Time :		15 minutes

EXAMPLE 3

Except the sequence (4), the other operations are same as in Example 1

(4)	$NiCl_2$. $6H_2O$	:	600 g/l
	H_3BO_3	:	30 g/l
	Polyvinyl alcohol	:	5-10% by wt.
	pH	:	2.1-3.5 (Electrometric)
	Temperature	:	70°C
	Time :		10 minutes

Price : TWO RUPEES

The following are among the main advantages of this invention :

1 It avoids the use of toxic and poisonous copper cyanide strike solutions which are costlier.

2. Since nickel being doposited by immersion, the process does not reruire rectifier and other electrical accessories as in the case of strike plating.

3. Since it is an immersion plating, the coverage of the surface and thickness of the deposit are uniform.

4. Immersion nickel plated mild steel may be used as a substrate for the deposition of copper and chromium in that order in bi and trimetal printing plate industries.

Sd. **ILLEGIBLE**
ASSTT. PATENTS OFFICER
Council of Scientific & Industrial Research

Date this 27th day of Augu t, 1974.

COMPLETE SPECIFICATION

IMPROVEMENTS IN OR RELATING TO THE IMMERSION DEPOSITION OF NICKEL ON MILD STEEL FOR SUBSEQUENT DEPOSITION WITH COPPER

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH

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This is an invention by Balkunje Anantha Shenoi, Scientist, Ramachandran Subramanian, Scientist and Ramasubbu Venkatachalam, Senior Scientific Assistant, all employed in Central Electrochemical Research Institute, Karaikudi-623006 and all are Indian Nationals.

This invention relates to improvements in or relating to the immersion deposition of nickel on mild steel for subsequent deposition with copper.

Hitherto it has been proposed to directly deposit copper on mild steel from a copper cyanide strike solution or direct nickel on mild steel surface from Wattls nickel solution for subsequent deposition with copper from acid copper bath.

This is open to the objection that copper cyanide salt used in the strike solution is costlier and is poisonous and toxic in nature. So, it requires a high hygienic atmosphere that can be achieved by the use of special fumehoods and fans. An initial deposition of copper from cyanide strike solution or nickel from Watt's nickel solutions is necessarily given on mild steel surface before regular thicker copper deposit from an acid copper bath. For this, extra busbar connections and rectifier are needed which add to the cost of plating.

The main object of the present invention is to obviate these disadvantages by using immersion nickel deposit on mild steel surface before regular copper deposition from acid copper bath.

The main finding underlying the invention is to use the immersion nickel bath consisting of nickel chloride 400-700 g/l, boric acid 25-50 g/l and an organic addition agent with one or more alcoholic group or its polymerised product such as glycerol polyvinyl alcohol or glycol 75-125 g/l to get a uniform adherent deposit on mild steel which facilitates further plating on steel.

To these ends, the invention broadly consists in degreasing the polished mild steel and cathodically electrocleaning in an allaline cleaner at 60-70°C followed by washing, rinsing and immersion in immersion nickel plating bath for about 10-15 minutes as the case may be. After getting the immersion deposit, it is rinsed and taken to conventional acid copper bath for furthei plating. The copper deposit obtained on mild steel after immersion nickel is very adherent.

The following are the typical examples given tc illustrate the invention and not to limit the scope of the invention :

EXAMPLE 1

1. Degrease the polished surface of mild steel
2. Cathodic electroclean in an alkali cleaner in the temperature range 60-70°C with current density of 155 asf
3. Rinse
4. Immersion nickeling in the solution given below

$NiCl_2$ $6H_2O$	:	500 g/l
H_3BO_3	:	25 g/l
Glycerol	:	75 ml/l
pH	:	2.1-3.5 (electrometric)
Temperature	:	75°C
Time	:	10 minutes

5. Rinse

6. Copper in conventional acid copper bath of the composition given below :

$CuSO_4$ $5H_2O$	:	200 g/l
H_2SO_4	:	75 g/l
Glue	:	0.1 g/l

7. Rinse and dry

EXAMPLE 2

Except the sequence (4). the other operations are the same as in example I.

4	$NiCl_2$. $6H_2O$	:	700 g/l
	H_3BO_3	:	40 g/l
	Glycol	:	100 g/l
	pH	:	2.1–3.5 (electrometric)
	Temperature	:	80°C
	Time	:	15 minutes

EXAMPLE 3

Except the sequence (4), the other operations are the same as in Example 1.

4.	$NiCl_2$. $6H_2O$	:	600 g/l
	H_3BO_3	:	30 g/l
	Polyvinyl alccohol		50-100 g/l
	pH	:	2.1-3.5 (electrometric)
	Temperature	:	70°C
	Time	:	10 minutes

The following are among the main advantages of this invention :

1. It avoids the use of toxic and poisonous copper cyanide strike solutions.

2. Since nickel is deposited by immersion, the process does not require rectifier and other electrical accessories as in the case of strike plating.

3. Since it is an immmersion plating the coverage of the surface and thickness of the deposit are uniform.

WE CLAIM :

1. A process for immersion deposition of nickel on mild steel for subsequent deposition of copper consisis in treating the polished degreased and electrocleaned mild steel in the immersion nickel bath which consists of nickel chloride boric acid and an organic addition agent with one or more alcoholic groups or its polymerised product such as glycerol, polyvinyl alcohol or glycol.

2. A process as claimed in claim 1 for immersion deposition of nickel on mild steel for subsequent deposition of copper wherein the nickel immersion bath comprises nickel chloride 400-700 g/1, boric acid 20-50 g/l and organic addition agent with one or more alcoholic group or its polymerised product such as glycerol, polyvinyl alcohol or glycol 75-125 g/1.

3. A procsss as claimed in claims 1 and 2 for immersion deposition of nickel on mild steel for subsequent deposition of copper wherein the immersion bath is operated at 60-70°C with a pH of 2.1 to 3.5

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Date this 16th day of October, 1975.

Dipti Printing, Cal-67--150 Copies