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IMPROVED PROCESS FOR THE ELECTRODEPOSITION OF
IRONNICKEL ALLOYS COATINGS ON METAL SUBSTRATES "

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.

PRICE : TWO RUPEES

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This is an invention by BAIKUNGE ANANTHA SHENOI, Scientist, Mrs. MALATHY PUSHPAVANAM, Junior Technical Assistant, Mrs. VIDYALAKSHMI, Senior Laboratory Assistant, all are Indian national and are employed in the Central Electrochemical Research Institute, Karaikudi -6, India.

The invention relates to the improvements in or relating to the electrodeposition of bright Iron Nickel deposits.

Hitherto nickel has been used as one of the undercoat for decorative chromium deposits. This is open to the objection that because of the high cost of imported nickel anodes and nickel salts, the cost of production is high.

The object of this invention is to obviate these disadvantages by using Iron-nickel alloy deposits which consists of 20-30% Iron in the alloy. To these ends, the invention broadly consists in the development of a process for the electrodeposition of bright Iron nickel deposits. The bath composition is as follows :

Nickel sulfate ($\text{NiSO}_4 \cdot 6 \text{H}_2\text{O}$)	60-100	g/l
Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)	60-100	g/l
Boric acid	30-45	g/l
Sodium citrate	20-40	g/l
Ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$)	15-25	g/l

using a brightner composition of

1, 4 butyne diol	1 g/l - 2 g/l
Pyridine sulfonic acid	2 g/l - 4 g/l
Saccharin	1 g/l - 3 g/l
Lignin sulfonic acid	2 g/l - 3 g/l

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The bath works between 3-6 A/dm² with agitation and at 55-65°C. (The same brightner works for pure nickel baths also).

The following are the example given to illustrate the invention

EXAMPLE-1

Nickel sulfate(NiSO ₄ 6H ₂ O)	75 g/l
Nickel chloride(NiCl ₂ 6H ₂ O)	75 g/l
Boric Acid	45 g/l
Sodium citrate	30 g/l
Ferrous sulfate(FeSO ₄ 7H ₂ O)	15 g/l
Butyne diol	1 g/l
Pyridine sulfonic acid	2 g/l
Saccharin	1 g/l
Lignin sulfonic acid	2 g/l
	PH 3
Temperature	63°C
Agitation	air
% Fe in the deposit	25%
Current density	4.5 A/dm ²

Pure nickel and Iron anodes were used. The nickel to Iron anode area should be in the ratio of 8-10:1.

The reflectivity was 98-100% when measured with reference to vacuum coated silver mirror.

EXAMPLE-2

Nickel sulfate(NiSO ₄ 6H ₂ O)	60 g/l
Nickel chloride(NiCl ₂ 6H ₂ O)	90 g/l
Boric Acid	30 g/l
Sodium citrate	20 g/l
Ferrous sulfate(FeSO ₄ 7H ₂ O)	20 g/l
Butyne diol	1 g/l
Pyridine sulfonic acid	5 g/l
Saccharin	1.5g/l
Lignin sulfonate	1 g/l
	PH 3.2
Temperature	60°C

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agitation	air
% Iron in the deposit	30%
Ni:Fe anode ratio	10:1
Reflectivity	95%

EXAMPLE 3

Nickel sulfate($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$)	100 g/l
Nickel chloride($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)	60 g/l
Boric acid	45 g/l
Sodium citrate	30 g/l
Ferrous sulfate($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$)	15 g/l
Butyne diol	1 g/l
Pyridine sulfonic acid	1 g/l
Saccharin	1 g/l
Lignin sulfonate	1 g/l
pH	2.8
Temperature	60 ⁸⁰ °C
Current density	5 A/dm ²
agitation	air
Iron in the deposit	30%
Ni : Fe anode ratio	8 : 1
Reflectivity	98%

The following are among the main advantages of this invention :

Bright deposits equivalent to pure nickel deposits (when measured with reference to vacuum coated silver mirror) 25-30% savings in the cost of nickel ; Suitability of the same brightner to pure nickel baths also.

Dated this 14th Day of January 1977.

Sd/-
Asstt. Patents Officer,
Council of Scientific & Industrial
Research.

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THE PATENTS ACT, 1970

COMPLETE SPECIFICATION

(Section—10)

**IMPROVED PROCESS FOR THE ELECTRODEPOSITION OF IRON-
NICKEL ALLOY COATINGS ON METAL SUBSTRATES**

COUNCIL OF SCIENTIFIC & 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 Balkunge Anantha Shenoi, Scientist :
Mrs. Malathy Pushpavanam, Junior Technical Assistant and Mrs
. Vidya lakshmi, Senior Laboratory Assistant, all are Indian
nationals and are employed in the Central Electrochemical
Research Institute, Maraikudi - 623 006, India.

The invention relates to an improved process for the electro-deposition of bright iron nickel alloy coatings on metal substrates.

Hitherto nickel has been used as one of the undercoat for decorative chromium deposits. This is open to the objection that because of the high cost of imported nickel anodes and nickel salts, the cost of production is high.

The process involves the steps of mechanical polishing of the metal substrates, degreasing, alkaline cleaning, pickling and plating in an iron-nickel alloy bath. In the known processes it has been possible to obtain only bright nickel coatings and iron-nickel depositions have been carried out only as a thin film and it has not been possible to obtain coatings of desired thickness by electrodeposition.

The object of this invention is to obviate these disadvantages by using iron-nickel alloy deposits which consists of 20-30% iron in the alloy and to obtain coating of upto 30 microns thickness.

The main finding of the invention is the development of a process by modifying the bath composition anode area and new additives useful as brighteners so as to obtain coatings with a bright finish so as to substitute the pure nickel decorative finish. The development of improved process of this invention will save nearly 30% of the cost of production.

The present invention accordingly consists of ^{an} improved process for the electrodeposition of iron nickel alloy coatings on metal substrates ^{wherein the} ~~(comprising using an)~~ electrolytic bath consisting of nickel sulphate, nickel chloride, ferrous sulphate, sodium citrate, boric acid and additives like butynediol, pyridine sulphuric acid, saccharine and sodium salt of lignin sulphonic acid as brighteners using iron nickel dual anodes.

According to another feature of the invention the electrolytic bath used consists of 60-100 g/l of nickel sulphate, 60-100 g/l of nickel chloride, 30 - 45 g/l of boric acid, 10-25 g/l of ferrous sulphate, 30-45 g/l of sodium citrate and 0.8 - 2 g/l of butynediol, 1-5 g/l of pyridine sulphonic acid, 1-5 g/l of saccharin and 0.1 - 2 g/l of sodium salt of lignin sulphonic acid as brighteners.

According to a further feature of the invention the ^{nickel} iron dual anodes used have a ratio of 8-10 : 1 and the electrodeposition is carried out at a temperature of 55-65°C, pH of 2.8 - 3.2 and a current density of 30 - 70 A/dm² with air agitation.

According to another further feature of the invention the metal substrate used are steel, copper or brass and the electrolytic bath contains 75 g/l of nickel sulphate, 75 g/l of nickel chloride, 15 g/l ferrous sulphate, 30 g/l sodium citrate, 30 g/l of boric acid and 1 g/l of butynediol, 1 g/l of pyridine sulphonic acid, 4 g/l of saccharin and 1 g/l of sodium salt of lignin sulphonic acid added as brighteners.

According to a further preferred feature of the invention the electrolytic bath contains 100 g/l of nickel sulphate, 60 g/l of nickel chloride, 20 g/l of ferrous sulphate, 45 g/l of sodium citrate, 45 g/l of boric acid and 1 g/l of butynediol, 2 g/l of pyridine sulphonic acid, 3 g/l of saccharine and 1 g/l of sodium salt of lignin sulphonic acid added as brighteners.

Further more the iron nickel dual anode used has a ratio of 1:8

The invention is further illustrated with the following examples

Example 1

Nickel sulphate (NiSO ₄ 6H ₂ O)	:	75 g/l
Nickel chloride (NiCl ₂ 6H ₂ O)	:	75 g/l
Boric acid	:	45 g/l
Sodium citrate	:	30 g/l
Ferrous sulphate (FeSO ₄ 7 H ₂ O)	:	15 g/l

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Butyne diol	:	1 g/l
Pyridine sulphonic acid	:	2 g/l
Saccharin	:	4 g/l
Lignin sulphonic acid	:	2 g/l
Temperature	:	63°C
pH	:	3
Agitation	:	Air
Percentage of iron in the deposit	:	25%
Current density	:	4.5 A/dm ²
Nickel and iron anode ratio	:	10 : 1
Reflectivity	:	98 - 100%

The reflectivity was measured with reference to vacuum coated silver mirror.

Example 2

Nickel sulphate (NiSO ₄ 6H ₂ O)	:	60 g/l
Nickel chloride (NiCl ₂ 6H ₂ O)	:	90 g/l
Boric acid	:	30 g/l
Sodium citrate	:	20 g/l
Ferrous sulphate (FeSO ₄ H ₂ O)	:	20 g/l
Butyne diol	:	1 g/l
Pyridine sulphonic acid	:	5 g/l
Saccharin	:	2 g/l
Lignin sulphonic acid (Sodium salt)	:	2 g/l
pH	:	3.2
Temperature	:	60°C
Current density	:	3 A/dm ²
Agitation	:	Air
Percentage of iron in the deposit	:	30%
Nickel : iron ratio	:	9 : 1
Reflectivity	:	95%

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Example 3

Nickel sulphate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$)	:	100 g/l
Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)	:	60 g/l
Boric acid	:	45 g/l
Sodium citrate	:	30 g/l
Ferrous Sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$)	:	15 g/l
Butyne diol	:	1 g/l
Pyridine sulphonic acid	:	1 g/l
Lignin sulphonate (sodium salt)	:	1 g/l
pH	:	2.8
Temperature	:	68°C
Current density	:	5 A/dm ²
Agitation	:	Air
Percentage of iron in the deposit	:	30%
Nickel : iron ratio	:	8 : 1
Reflectivity	:	98%

WE CLAIM:

1) In an improved process for the electrodeposition of iron-nickel alloy coatings on metal substrates ^{wherein} ~~the electrolytic bath~~ ^{using the} electrolytic bath consisting of nickel sulphate, nickel chloride, ferrous sulphate, sodium citrate, boric acid and additives like butyne/diol, pyridine, a sulphonic acid, saccharine and sodium salt of lignin sulphonic acid as brighteners using iron nickel dual anodes.

2) Process as claimed in claim 1 wherein the electrolytic bath contains 60 - 100 g/l of nickel sulphate, 60 - 100 g/l of nickel chloride, 30-45 g/l of boric acid, 10 - 25 g/l of ferrous sulphate, 30 - 45 g/l of sodium citrate and 0.8 - 2 g/l of butyne-diol, 1-5 g/l of pyridine sulphonic acid, 1-5 g/l of saccharin and 0.1 - 2 g/l of sodium salt of lignin sulphonic acid as brighteners.

- 3) Process as claimed in claims 1 and 2 wherein the iron dual anodes used have a ratio of 8-10 : 1 and the electrodeposition is carried out at a temperature of 55-65°C, pH of 2.8 - 3.2 and a current density of 30 - 70 A/dm² with air agitation.
- 4) Process as claimed in claims 1 to 3 wherein the metal substrates used are steel, copper or brass and the electrolytic bath contains 75 g/l of nickel sulphate, 75 g/l of nickel chloride, 15 g/l of ferrous sulphate, 30 g/l of sodium citrate, 30 g/l of boric acid and 1g/l of butyne diol, 1 g/l of pyridine sulphonic acid, $\frac{4}{2}$ g/l of saccharine and 1 g/l of sodium salt of lignin sulphonic acid added as brighteners.
- 5) Process as claimed in any of the preceding claims wherein the electrolytic bath contains 100 g/l of nickel sulphate, 60 g/l of nickel chloride, 20 g/l of ferrous sulphate, 45 g/l of sodium citrate, 45 g/l of boric acid and 1 g/l of butyne diol, 2 g/l of pyridine sulphonic acid 3 g/l of saccharine and 1 g/l of sodium salt of lignin sulphonic acid added as brighteners.
- 6) Process as claimed in claims 1 and 3 wherein the iron nickel dual anode used has a ratio of 1:8
- 7) Improved process for the electrodeposition of iron nickel alloy coatings on metal substrate substantially as herein described and illustrated.

Dated this 18th day of March, 1978

h. n. n. n. n.

Scientist (Patents)
Council of Scientific & Industrial Research