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PROVISIONAL SPECIFICATION

IMPROVEMENTS IN OR RELATING TO ELECTRODEPOSITION OF IRON-CHROMIUM-NICKEL ALLOY DEPOSITS

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), (Miss) SETHURAMAN GOWRI (Junior Scientific Assistant) and (Miss) PANAKKAL LAZER ELSIE (Senior Laboratory Assistant). all are citizens of India and employed in the Central Electrochemical Research Institute, Karaikudi-3, S. Rly, Madras, India.

This invention relates to improvements in or relating to Electrodeposition of Iron-Chromium-Nickel alloy deposits.

Hitherto it has been proposed to electrodeposit the Iron-Chromium-Nickel alloy deposits from baths containing (a) urea, (b) EDTA, (c) alum and (d) chlorides.

This is open to the objection that the baths give dull deposits with basic inclusions which require polishing after the plating and the baths do not give satisfactory deposits continuously.

The object of this invention is to obviate these disadvantages by using an aqueous solution containing the suitable salts of the metals, citrate, boric acid, addition agent such as formaldehyde or its derivative etc.

To these ends, the invention broadly consists in electrolysing an aqueous solution containing suitable salts of metals, complexing agent such as citrate, addition agent etc. with platinum or stainless steel as anode and copper, brass as cathodes.

The following typical examples are given to illustrate the invention:

EXAMPLE 1

Chromium sulphate	300—350 gms per litre
Nickel formate or sulphate	: 35—40 ..
Ferrous sulphate	: 25—30 ..
Trisodium citrate	: 100—120 ..
Boric acid	: 25—40°C ..
Ammonium sulphate	: 50—100 ..
Sodium fluoride	: 6—8 ..
Glycine	: 7—20 ..
Addition agent	: 5—20 ..
Temperature	: 25—40°C
Time	: 3—30 minutes
Current density	: 9—32 amp/dm ²

The deposit contains 10—11% nickel, 25—35% chromium and 55—65% iron.

EXAMPLE 2

Chromium-potassium sulphate	:	400—550 gms per litre
Nickel formate or sulphate	:	35—40 ..
Ferrous sulphate	:	25—30 ..
Trisodium citrate	:	100—120 ..
Boric acid	:	30—50 ..
Sodium fluoride	:	8—10 ..
Addition agent	:	18—35 ..
Temperature	:	30—50°C
Time	:	3—30 minutes
Current density	:	10—30 amp/dm ²

The deposit contains 10—13% nickel, 18—35% chromium and 50—70% iron.

The following are among the main advantages of the invention:

1. It is relatively a cold process.
2. The deposit is bright in the as-plated condition and free from basic inclusions.
3. The solution continuously gives uniform deposit without blackening at the edges.
4. The operating conditions are less critical and easy to control.
5. The conductivity of the solution is good.
6. No need of heat treatment in vacuum after deposition to improve the adhesion.
7. The efficiency is comparable with the known baths.
8. The deposit can be grown to sufficient thickness without any change in the ductility of the deposit.

Dated this 4th day of March, 1968.

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COMPLETE SPECIFICATION

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED
BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT

(ACT XXI OF 1960)

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, MISS SETHURAMAN GOWRI Junior Scientific Assistant and MISS PANAKKAL LAZER ELSIE, Senior Laboratory Assistant, all of the Central Electrochemical Research Institute, Karai-kudi-3, S. Rly., Madras, India, all Indian citizens.

This invention relates to improvements in or relating to Electrodeposition of Iron-Chromium-Nickel alloy Deposits and has particular reference to electrodeposition of iron-chromium-nickel alloy and is related to metal finishing industry.

Hitherto it has been proposed to electrodeposit iron-chromium-nickel alloy from sulphate baths containing complexing agents such as (a) urea, (b) EDTA, (c) alum and (d) chlorides.

Hitherto known processes are open to the objection that the baths give dull deposits with basic inclusions (which increase the internal stress of the deposit) and which give only mottled appearance after polishing.

The object of the invention is to obviate these disadvantages by using a bath which would give deposit free from basic inclusions.

The principle underlying the invention involves the electrodeposition of alloy of iron, chromium and nickel from a bath containing the metals in the form of citrate complexes with suitable addition agents.

The investigation leads to the deposition of ternary alloy from baths containing chromium sulphate and chrome alum, nickel formate and ferrous sulphate. The use of nickel formate, as the source of nickel, avoids many difficulties encountered with the nickel sulphate. By using nickel sulphate as one of the constituents, it is not possible to adjust the concentration of nickel within the required range, since nickel sulphate forms a less soluble nickel ammonium sulphate by combining with ammonium sulphate.

According to the present invention, the bath for electrodeposition of iron-chromium-nickel alloy comprising chromium sulphate, ferrous sulphate, a nickel salt and a complexing agent such as trisodium citrate is characterised in that the nickel salt consists of nickel formate.

Addition agents such as ammonium sulphate, boric acid, sodium fluoride and glycine are added to the bath. Thus, hexamine is added to the bath to get a bright deposit.

The bath is suitable for the electrodeposition of iron-chromium-nickel alloy deposits containing 10—11% nickel, 25—33% chromium and 55—65% iron.

The process for electrodeposition using the bath consists in electrolysing an aqueous solution containing sulphates of chromium and iron, nickel formate and trisodium citrate with addition agents such as ammonium sulphate, boric acid, sodium fluoride and glycine and hexamine.

The alloy of iron, chromium and nickel is electrodeposited from the bath containing the metals in the form of citrate complexes with the addition agents.

Thus the ternary alloy is deposited from the bath containing chromium sulphate and chrom alum, nickel formate and ferrous sulphate.

Platinum or preferably, stainless steel, is used as the anode and copper, brass, mild steel or aluminium as cathodes.

A bright deposit of iron, nickel and chromium alloy that is resistant to immersion in 5% sulphuric acid and 3% sodium chloride is obtained.

The deposit is completely free from basic inclusions (as examined under the microscope). The internal stress of the deposit is low (33—35 kg/mm²) on copper than on brass, mild steel, nickel etc. Buffer capacity of the bath is commendable. 4-5 microns thick alloy deposit is resistant to 5% sulphuric acid (greater than 115—120 hours immersion) and 3% sodium chloride (greater than 450—500 hrs. immersion). Current efficiency of the alloy deposition is comparable (30—35%) with that of the existing baths (23—27%). Platinum or stainless steel is used as the anode and copper, brass, mild steel and aluminium as cathodes.

Thus, an aqueous solution containing sulphates of chromium and iron, nickel formate, trisodium citrate, ammonium sulphate, boric acid, glycine and sodium fluoride is electrolysed. Addition agents, such as formaldehyde, hexamine improve the appearance of the deposit.

The following typical examples are given to illustrate the invention and the usual preplating treatments are sufficient to get good quality deposit.

EXAMPLE 1

Chromium sulphate	..	300 gms per litre
Nickel formate	..	40 " "
Ferrous sulphate	..	25 " "
Trisodium citrate	..	100 " "
Boric acid	..	30 " "
Ammonium sulphate	..	50 " "
Sodium fluoride	..	8 " "
Glycine	..	10 " "
Addition agent such as Hexamine	..	18 " "
Temperature	..	30°C
Time	..	5 minutes
pH	..	1.8
Current density	..	18.6 amp/dm ²
Current efficiency	..	30—35%

The deposit contains 10-11% nickel, 25—35% chromium and 50—65% iron.

EXAMPLE 2

Chromium-potassium sulphate	..	400 gms per litre
Nickel formate	..	40 " "
Ferrous sulphate	..	25 " "
Trisodium citrate	..	100 " "
Boric acid	..	30 " "
Sodium fluoride	..	10 " "
Addition agent such as Hexamine	..	25 " "
Temperature	..	30°C

Time	..	5 minutes
pH	..	1.8
Current density	..	18.6 amp/dm ²
Current efficiency	..	30—35%

The deposit contains 10—13% nickel, 18—35% chromium and 50—70% iron.

An alloy of iron, chromium and nickel deposited from the abovementioned new formulation is uniform, adherent, coherent and free from basic inclusions and bright which avoids polishing after plating. The conductivity of the bath is comparable with hitherto known baths and the operating conditions are less critical and easy to control. The deposit is ductile and needs no heat treatment in vacuum after deposition to improve adhesion.

The alloy approximately contains 10-11% nickel, 25—35% chromium, 55—65% iron. The alloy deposit is resistant to 5% sulphuric acid and 3% sodium chloride for a considerable period. The efficiency of the deposition is comparable with the known baths or even higher. Both platinum and stainless steel anodes are used. With platinum anode, after the passage of 36 amp. hr./litre of electricity, the nickel content increases to 20% which brings down the efficiency of alloy deposition. So, at this stage, the bath is corrected to the initial composition after analysis. With 304 stainless steel anode, the rates of anodic and cathodic efficiency are same and hence, there is no much change in the efficiency and composition of the alloy with time. The bath should be corrected to the initial composition after the passage of 48—60 amp. hrs./litre electricity. Stainless steel anode is found to be convenient, efficient and cheap compared to platinum anode. The new formulation is quite convenient for depositing an alloy of iron chromium and nickel with any desired composition.

The following are among the main advantages of the invention:

1. The bath is operated at room temperature (30—35°C).
2. The deposit is bright in the as-plated condition and free from basic inclusions.
3. The solution continuously gives uniform deposit without blackening at the edges.
4. The operating conditions are less critical and easy to control.
5. The conductivity of the solution is good.
6. No need of heat treatment in vacuum after deposition to improve the adhesion, as it is done in the hitherto known process.
7. The efficiency is comparable with the known bath.
8. The deposit can be grown to sufficient thickness (up to 25 μ) without any change in the ductility of the deposit.

We claim:

1. A bath for electrodeposition of iron-chromium nickel alloy comprising chromium sulphate, ferrous sulphate, a nickel salt and a complexing agent such as trisodium citrate, characterised in that the nickel salt consists of nickel formate.

2. A bath as claimed in claim 1 wherein addition agents such as ammonium sulphate, boric acid, sodium fluoride and glycine are added to the bath.

3. A bath as claimed in Claim 1 or 2 wherein hexamine is added to the bath.

4. A bath for the electrodeposition of iron-chromium-nickel alloy deposits containing 10-11% nickel, 25—35% chromium and 55—65% iron substantially as hereinbefore described.

5. A process for electrodeposition using the bath as claimed in Claim 1 which consists in electrolysing an aqueous solution containing sulphates of chromium and iron, nickel formate and trisodium citrate.

6. A process as claimed in claim 5 wherein addition agents such as ammonium sulphate, boric acid, sodium fluoride and glycine are added to the aqueous solution.

7. A process as claimed in Claim 5 or 6 wherein hexamine is added to the bath.

8. A process as claimed in any of the preceding Claims 5 to 7 wherein the alloy of iron, chromium and nickel is electrodeposited from the bath containing the metals in the form of citrate complexes with the addition agents.

9. A process as claimed in any of the preceding Claims 5 to 8 wherein the ternary alloy is deposited from the bath containing chromium sulphate and chrome alum, nickel formate and ferrous sulphate.

10. A process as claimed in any of the preceding claims 5 to 9 wherein stainless steel is used as the anode.

11. A process as claimed in any of the preceding Claims 5 to 10 wherein platinum or preferably stainless steel, is used as the anode and copper, brass, mild steel or aluminium as cathode.

12. A process as claimed in any of the preceding claims wherein a bright deposit of iron-nickel and chromium alloy that is resistant to immersion in 5% sulphuric acid and 3% sodium chloride is obtained.

13. A process for electrodeposition of iron-chromium-nickel alloy deposits substantially as described in the examples.

Dated this 30th day of December, 1968.

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