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Index at acceptance—70C4 [LVIII(5)].

### Provisional specification

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

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 1860).

*The following specification describes the nature of this invention:*

This is an invention by BALKUNJE ANANTHA SHENOI (Scientist) Miss PANAKKAL LAZAR ELSIE (Senior Laboratory Assistant) and Miss SETHURAMAN GOWRI (Junior Scientific Assistant) all are citizens of India and employed in the Central Electrochemical Research Institute, Karaikudi-3, India.

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

Hitherto it has been proposed to electrodeposit the Iron-chromium alloy deposits from baths containing (a) urea (b) fluoroborate (c) trivalent chromium salt (d) glycine.

This is open to the objection that the baths give deposits with basic inclusions and the deposits are not resistant to 5% sulphuric acid, operated at high current density and temperature.

The object of this invention is to obviate these disadvantages by using an aqueous solution containing the suitable salts of the metals, boric acid etc.

To these ends, the invention broadly consists in electrolysing an aqueous solution containing the sulphates of metals, complexing agent such as citrate, ammonium sulphamate or ammonium sulphate with platinum or stainlesssteel as anode and copper brass as cathodes.

The following typical examples are given to illustrate the invention:

#### Example I

Chromium-potassium sulphate	. 500-550 gms/lit
Ferrous sulphate	. 20-25 gms/lit.
Trisodium citrate	. 100-150 gms/lit
Boric acid	. 30-50 gms/lit
Ammonium sulphamate	. 50-100 gms/lit
Sodium fluoride	. 8-10 gms/lit
Temperature	. 25-40°C.
Current density	. 5-28 amp/dm <sup>2</sup>
Time	. 3-30 minutes

The deposit contains 40-45% chromium and 65-60% iron.

#### Example II

Chromium sulphate	. 400-420 gms/lit
Ferrous-ammonium sulphate	. 20-35 gms/lit
Trisodium citrate	. 100-120 gms/lit
Boric acid	. 50 gms/lit
Ammonium sulphate.	. 75-100 gms/lit
Sodium fluoride	. 7-12 gms/lit
Temperature	. 30-60°C
Current density	. 6-30 amp/dm <sup>2</sup>
Time	. 4-30 minutes

The deposit contains 35-45% chromium and 55-65% iron.

The following are among the main advantages of the invention:

1. It is relatively a cold process.
2. The deposit is uniform and free from basic inclusions.
3. The operating conditions are less critical and easy to control.
4. The conductivity of the solution is good.
5. The deposit can be grown to sufficient thickness without changing the ductility of the deposit.
6. The deposit is resistant to 5% sulphuric acid (over 65 hours immersion) and 3% sodium chloride (over 250 hours immersion).

Dated this 2nd day of March, 1968.

PATENTS OFFICER,

Council of Scientific and Industrial Research.

### COMPLETE SPECIFICATION

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

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 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, MISS PANAKKAL LAZAR ELSIE, Senior Laboratory Assistant and MISS SETHURAMAN GOWRI, Junior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, India all Indian citizens.

This invention relates to Improvements in or relating to Electrodeposition of Iron-Chromium Alloy Deposits and has particular reference to electrodeposition of iron-chromium alloy and is related to Metal finishing industry.

Hitherto it has been proposed to electrodeposit the iron-chromium alloy deposits from baths containing (a) urea, (b) fluoborate, (c) trivalent chromium salt and (d) glycine.

Hitherto known processes are open to the objection that the baths give deposits with basic inclusions and/are operated at high current density and temperature, and the deposits are not resistant to 5% sulphuric acid.

The object of this invention is to obviate these disadvantages by using an aqueous solution containing sulphates of metals, Boric acid and complexing agent such as citrate etc.

The principle underlying the invention involves the electrodeposition of an alloy of iron and chromium, from a bath containing the metals in the form of citrate complexes.

The new plating bath utilises potassium chromium alum, which is cheaper (Rs. 4/500 gms) than chromium sulphate (Rs. 62/500 gms) which is used in hitherto known processes. The deposit is completely free from basic inclusions (as examined under the microscope). The internal stress of the deposit is low on copper substrate (25 to 27/kg/mm<sup>2</sup>) than on brass, mild steel, nickel etc. The buffer capacity of the bath is commendable. 2.5 to 3 microns thick alloy deposit is resistant to 5% sulphuric acid (65 to 70 hrs) and 3% sodium chloride (250 to 300 hrs immersion). Current efficiency of the alloy deposition is comparable (25-26%) with that of the existing baths (20-6%).

According to the present invention the process for the electrodeposition of iron-chromium alloy deposits containing 40-45% chromium and 60-55% iron consists in electrolysing an aqueous solution containing sulphates of chromium (500-550 g/l) iron (20-25 g/l), complexing agent such as citrate (100-150 g/l), ammonium salt (50-100 g/l), boric acid (30-50 g/l) and sodium fluoride (8-10 g/l) with platinum or stainless steel as anode and copper, brass, mild steel, aluminium etc. as cathode operating at the current densities between 5-28 amp/dm<sup>2</sup> at temperatures between 25-40°C.

A deposit that can withstand immersion in 5% sulphuric acid for over 65-70 hrs. and 3% sodium chloride for over 250-300 hrs is obtained.

Thus, an aqueous solution containing sulphates of metals, (e.g.) chromium potassium sulphate, ferrous sulphate, complexing agent such as trisodium citrate, ammonium salt (sulphamate or sulphate), boric acid and sodium fluoride is electrolysed.

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

#### Example 1

Chromium-Potassium sulphate	. 500-550 gms/litre
Ferrous sulphate	. . . 20-25 "
Trisodium citrate	. . . 100-150 "
Boric acid	. . . 30-50 "
Ammonium sulphamate	. . . 50-100 "
Sodium fluoride	. . . 8-10 "
Temperature	. . . 25-40°C
Current density	. . . 5-28 amp/dm <sup>2</sup>
Time	. . . 3-30 minutes
pH	. . . 1.6-2.5
Current efficiency	. . . 25-26%

The deposit contains 40-45% chromium and 55-60% iron.

#### Example 2

Chromium Potassium sulphate	. 400-420 gms/litre
Ferrous ammonium sulphate	. 20-35 "
Trisodium citrate	. . . 100-120 "
Boric acid	. . . 50 "
Ammonium sulphate.	. . . 75-100 "
Sodium fluoride	. . . 7-12 "
Temperature	. . . 30-45°C
Current density	. . . 6-30 amp/dm <sup>2</sup>
Time	. . . 4-30 minutes
pH	. . . 1.6-2.5
Current efficiency	. . . 24-26%

The deposit contains 35-45% chromium and 55-65% iron.

#### Advantages

1. The bath is operated at room temperature (30-35°C).
2. The deposit is uniform and free from basic inclusions.
3. The operating conditions are less critical and easy to control.
4. The conductivity of the solution is good.
5. The deposit can be grown to sufficient thickness (25 microns) without change in the ductility of the deposit.
6. The deposit is resistant to 5% sulphuric acid (over 65 to 70 hrs immersion) and 3% sodium chloride (over 250-300 hrs. immersion).

An alloy of iron and chromium deposited from the above mentioned new plating bath with 5-28 amp/dm<sup>2</sup> at 25-40°C is uniform adherent, coherent and free from basic inclusions. The conductivity of the bath is comparable and the operating conditions are less critical and easy to control. The deposit can be grown to sufficient thickness without change in the ductility of the deposit. The alloy approximately contains 40 to 45% chromium, 60 to 55% iron. The buffer capacity of the bath is quite good and the alloy deposit withstands immersion in 5% sulphuric acid and 3% sodium chloride for a considerable period. The current efficiency of the alloy is comparable with that of the known baths. The deposit requires no post-treatment such as buffing. The composition and the efficiency of alloy deposition do not change much with time. However, the bath should be corrected to the usual composition after 35-40 amp. hrs/litre. Stainless steel anode is found to be more convenient and efficient compared to platinum anode. The new plating bath is quite suitable for getting an alloy of iron and chromium with any desired composition.

#### We claim:

1. A process for the electrodeposition of iron-chromium alloy deposits containing 40-45% chromium and 60-55% iron which consists in electrolysing an aqueous solution containing sulphates of chromium (500-550 g/l) iron (20-25 g/l), complexing agent such as citrate (100-150 g/l), ammonium salt (50-100 g/l), boric acid (30-50 g/l) and sodium fluoride (8-10 g/l) with platinum or stainless steel as anode and copper, brass, mild steel, aluminium etc. as cathode operating at the current densities between 5-28 amp/dm<sup>2</sup> at temperatures between 25-40°C.
2. A process as claimed in Claim 1 which gives a deposit that can withstand immersion in 5% sulphuric acid for over 65-70 hrs and 3% sodium chloride for over 250-300 hrs.

3. A process as claimed in claims 1 and 2 employing stainless steel or platinum as anode.

4. A process for the electrodeposition of iron-chromium alloy deposits containing 40-45% chromium

and 60-55% iron substantially as hereinbefore described

Dated this 30th day of December, 1968.

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