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“IMPROVEMENTS IN OR RELATING TO THE PREPARATION OF ELECTROLYTIC CHROMIUM POWDER”

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH.,

REFI MARG, NEW DELHI-I, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT. (ACT XXII OF 1860.)

The following specification describes the nature of this invention :—

This is an invention by Rengachari Srinivasan, Scientist ; Krishnan Naganathan, Senior Scientific Assistant ; Pitchaimuthu Adaikkalam, Senior Laboratory Assistant ; Periyaiyah Ramachandran, Senior Laboratory Assistant ; veeraraghava Aravamuthan, Scientist, all Indians and employed in Central Electrochemical Research Institute, Karaikudi.

This invention relates to improvements in or relating to the preparation of chromium powder by electrochemical methods.

Hitherto it has been proposed to prepare the chromium powder by aluminothermic process and comminute the resulting chromium metal to powder, or produce chromium flakes by electrolysis of chromium ammonium alum and then comminute it to produce chromium powder.

This is open to the objection that the powder prepared by aluminothermic process is only 97% pure which makes them unsuitable for welding electrode preparation. The process based on electrolysis of chromium ammonium alum involves a number of evaporation and crystallisation steps as well as rather critical operating conditions in electrolysis with respect to pH, ammonia concentration, current concentration etc., Also the carbon content of the produce is 0.025% while the specifications call for a carbon content of not more than 0.01%. The purity of the metal obtained from chrome alum bath is 98.5 to 99.0%.

The object of this invention is to obviate these disadvantages by preparing the chromium electrolytically from chromic acid and powdering it so that the purity is not less than 98.5% which is suitable for welding electrodes manufacture.

To these ends, the invention broadly consists in depositing chromium in the form of brittle sheets from chromic acid containing known amount of sulphate ion on copper coated stainless steel cathodes and then powdering the chromium deposit after removing the chromium deposit along with copper under coat and dissolving away the copper in nitric acid.

The following typical examples are given to illustrate the invention :—

Example I

Cathode	: Electrolytically copper coated stainless steel
Anode	: Lead-silver (1%) alloy
Bath	: 250 gpl of chromic acid solution containing 2.5

Example I Contd.

	: gpl of sulphuric acid
Current density	: 32 amperes per sq. dm.
Temperature	: 40°C
Duration	: 48 hrs
Current efficiency	: 18%
Purity	: 98.7%

Example—II

Cathode	: Copper coated stainless Steel
Anode	: Lead-silver (1%) alloy
Bath	: 250 gpl of chromic acid solution containing 2.5 gpl of sulphuric acid
Current density	: 24 amperes per sq. dm.
Temperature	: 40°C
Duration	: 24 hrs
Current efficiency	: 18%
Purity	: 99.3%

The carbon content of the metal was 0.006% in both cases.

The following are among the main advantages of the invention. The chromium metal obtained is not less than 98.5% pure. The deposition of the metal on copper coated stainless steel makes it easier to obtain uniform deposits and to remove it from the stainless steel base as flakes along with copper. Subsequently copper is dissolved in nitric acid. This procedure has the advantage of minimum dissolution of copper in the recovery of 98% purity electrolytic chromium. Since the deposit is obtained in the form of flakes it can be ground to the required mesh size.

S/d. Illegible

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Council of Scientific and Industrial Research.

Dated this 21st day of June, 1973.

Pices : TWO RUPEES

THE PATENTS ACT 1970

Complete Specification No. 138312

Section 10

“IMPROVEMENTS IN OR RELATING TO THE PREPARATION OF
ELECTROLYTIC CHROMIUM POWDER”

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH RAFI MARG.

NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE
REGISTRATION OF SOCIETIES ACT (ACT XXII 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 Rengachari Srinivasan, Scientist ; Krishnan Naganathan, Senior Scientific Assistant ; Pitchaimuthu Adaikkalam, Senior Laboratory Assistant ; Periyaiyah Ramachandran, Senior Laboratory Assistant ; Veeraraghava Aravamuthan, Scientist, all Indians and employed in Central Electrochemical Research Institute, Karaikudi.

This invention relates to improvements in or relating to the preparation of electrolytic chromium powder for the manufacture of welding electrodes.

Hitherto it has been proposed to prepare the chromium powder by aluminothermic process and comminute the resulting chromium metal to powder, or produce chromium flakes by electrolysis of chromium ammonium alum and then comminute it to produce chromium powder.

This is open to the objection that the powder prepared by aluminothermic process is only 97% pure which makes it unsuitable for welding electrode preparation. The process based on electrolysis of chromium ammonium alum involves a number of evaporation and crystallisation steps as well as rather critical operating conditions in electrolysis with respect to pH, ammonia concentration, current concentration, etc. Also the carbon content of the product is 0.025% while the specifications call for a carbon content of not more than 0.01%.

The object of this invention is to obviate these disadvantages by preparing the chromium electrolytically from chromic acid and powdering it so that the purity is not less than 98.5% which is suitable for welding electrodes manufacture.

The invention broadly consists in depositing chromium in the form of brittle sheets from chromic acid containing known amount of sulphate ion on copper coated stainless steel cathodes and then powdering the chromium deposit after removing the copper substrate in nitric acid.

The chromium deposit obtained is easily peeled off as a brittle sheet along with a thin undercoat of copper of 3 to 4 thou thickness which after treatment with nitric acid yields bright flaky deposits of chromium after dissolution of the copper portion. Massive sheets of copper need not therefore be used as the starting sheets and dissolved out.

According to the present invention, there is provided a process for making electrolytic chromium powder which consists of an initial step of electro-deposition of chromium using a solution of chromic acid containing 250-300 gpl chromic acid and 2.5-3 gpl

sulphuric acid as electrolyte at a temperature of 40-50°C with an anode of lead silver (about 1%) alloy and a copper coated stainless steel cathode, the copper coating being 3-4 thou in thickness, electro-deposition of chromium is conducted in a range of cathodic current density of 15-40 amps/sq. dm. preferably 20-30 amps/sq. dm., the area of cathode to area of anode being around 1 : 3 followed by recovery of deposited chromium as flakes by peeling of copper and chromium together from the stainless steel and dissolving the copper in nitric acid, chromium metal flakes of not less than 98.5% purity thus obtained are powdered to desired sizes by mechanical means.

The process employs an electrolysing step wherein are used a solution of chromic acid containing 250 to 300 gpl of chromic acid and 2.5 to 3.0 gpl of sulphuric acid as the electrolyte at a temperature of 40 to 45°C and a cathode made of stainless steel initially given a coat of copper of 3 to 4 thou thickness which facilitates a smooth and coherent deposit of chromium and which is later removed along with the chromium and after treatment with nitric acid or a mixture of chromic acid and sulphuric acid enables recovery of chromium of not less than 98.5% purity, the current density employed being in the range of 15 to 40 A/dm² at the cathode, preferably 20 to 30 A/dm² and the cathode to anode ratio employed being around 1:3.

Being a simple process, no flow sheet and accompanying description are considered necessary.

EXAMPLE—I

Cathode	Electrolytically copper coated stainless steel (CuSO ₄ - about 200 gpl H ₂ SO ₄ - about 25 gpl c.d. - 2.5 A/dm ²)
Anode	: Lead-Silver (1%) alloy
Bath	: 250 gpl of chromic acid solution containing 2.5 gpl of sulphuric acid
Current density	: 30 A/dm ²
Temperature	: 40°C

EXAMPLE—I (contd.)

Duration	: 48 hrs
Current efficiency	: 18%
Purity	: 98.7%

EXAMPLE—II

Cathode	: Copper coated stainless steel as given in Example—I
Anode	: Lead-Silver (1%) alloy
Electrolyte	: 300 gpl chromic acid containing 3.0 gpl sulphuric acid
Current density	: 22 A/dm ²
Temperature	: 40°C
Duration	: 24 hrs
Current efficiency	: 19%
Purity	: 98.9%

EXAMPLE—III

Cathode	: Copper coated stainless steel as given in Example—I
Anode	: Lead-Silver (1%) alloy
Electrolyte	: 250 gpl chromic acid containing 2.5 gpl sulphuric acid
Current density	: 30 A/dm ²
Temperature	: 43°C
Duration	: 24 hrs
Current efficiency	: 18%
Purity	: 99%

The following are among the main advantages of the invention. The chromium metal obtained is not less than 98.5% pure. The deposition of the metal on copper coated stainless steel makes it easier to obtain uniform and compact deposits and to remove it from the stainless steel base. This procedure has the advantage of minimum dissolution of copper in the recovery of 98% purity electrolytic chromium. Since the deposit is obtained in the form of flakes it can be ground to the required mesh size.

The invention consists of an electrolytic method followed by a grinding procedure. The electrolytic method makes use of a simple procedure of pre-coating the cathode with a metal (copper) which enables the formation of a smooth over-layer of chromium deposit and an electrolyte (chromic acid-sulphuric acid) which gives a pure deposit of chromium. The initial deposit of copper is later removed by treatment with nitric acid or a mixture of chromic acid and sulphuric acid and this treatment does not affect the purity of the deposited chromium. A thin deposit of copper onto stainless steel is sufficient and massive copper sheet need not be used as the starting sheet. The chromium is obtained as brittle flakes which can be easily powdered. Being an electrolytic method, the purity of the product can be consistently maintained by ensuring the electrolyte does not get contaminated and scaling up of production by multiplication of cells is possible.

WE CLAIM :

1. A process for making electrolytic chromium powder which consists of an initial step of electrodeposition of chromium using a solution of chromic acid containing 250-300 gpl chromic acid and 2.5-3 gpl sulphuric acid as electrolyte at a temperature of 40-50°C with an anode of lead silver (about 1%) alloy and a copper coated stainless steel cathode, the copper coating being 3-4 thou in thickness, electro-deposition of chromium is conducted in a range of cathodic current density of 15-40 amps/sq. dm, preferably 20-30 amps/sq.dm., the area of cathode to area of anode being around 1:3 followed by recovery of deposited chromium as flakes by peeling of copper and chromium together from the stainless steel and dissolving the copper in nitric acid, chromium metal flakes of not less than 98.5% purity thus obtained are powdered to desired sizes by mechanical means.

R. Bhaskar Pai

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Dated this 25th day of June, 1974