Patented Sept. 29, 1936 2,055,963

UNITED STATES PATENT OFFICE

2,055,963

MAKING THICK DEPOSITS OF ELECTROLYTIC CHROMIUM AND THE SEPARATION THEREOF FROM THE CATHODE SURFACE

John W. Boss, Livingston, Mont.

No Drawing. Application October 2, 1935, Serial No. 43,288

7 Claims. (Cl. 204—1)

The present invention relates to metallic chromium and a process for making the same by electrolysis and more particularly to depositing a thick layer of chromium metal from aqueous solutions of chromium compounds and subsequently separating the deposited metal from the cathode surface.

An object of the invention is to provide means for depositing thick layers of chromium on a cathode by electrolysis from aqueous solutions of chromium compounds.

A further object is to prepare a cathode which will permit obtaining thick coats of chromium. Another object is to provide means for removing the deposited chromium from the cathode and a still further object is to provide a method for treating the cathode with the deposit to cause separation of the deposited metal from the cathode.

In the prior art of chromium plating, it is not customary to remove in metallic form any plate made. Metallic chromium for use in the arts has generally been prepared by pyro-metallurgical means. This entailed the use of very high temperatures and required expensive equipment and complex operation.

In the electrolytic method of chromium preparation from aqueous solutions of chromium compounds there are several difficulties, namely, the tendency of the deposited chromium to crack and separate from the cathode surface before a thickness of 0.01 inch is reached. Also much difficulty is encountered in complete removal of a deposit of electrolytic chromium when it is secured.

The present invention provides a means whereby electrolytic chromium may be deposited on a cathode in sufficient thickness to be commercially valuable to users of chromium metal, that is, to prevent peeling at any time during deposition and to provide satisfactory and economical means of stripping such thick deposits from the underlying surface.

In order to obtain a deposit of sufficient thickness, an especially prepared cathode is employed. The only practical substance for this cathode has been found to be tin or alloys of tin. It is necessary to etch the surface of this cathode material by any of many known means, such as immersion in sulphuric acid of specific gravity 1.70. The etching is carried out until the surface of the tin has become visibly changed. At this point the cathode is rinsed in acid of half the above strength to remove sulphides that may have been formed and finally rinsed in water to remove all acid and arrest further action. A satisfactory etched tin surface is of a light gray color with a crystal structure often visible to the naked eye. The etching should not be carried to such a point as to cause surface irregularity in the cathode or the chromium will be roughly deposited. It should be noted that tin without etching is not sufficient to secure thick deposits; neither do other etched metals answer the purpose because the deposited chromium cannot be readily removed therefrom.

When such an etched tin surface is made the cathode in a bath such as the well known "Sargent's solution" an electrolytic deposit of chromium may be built up to a thickness limited only by the construction of the cell or by progressive roughening or "treeting" of the deposit. When a tin surface is prepared as described and a deposit of electrolytic chromium made thereon, and the chromium removed as subsequently described it is not necessary to etch the tin surface thereafter. Repeated deposits may be made and stripped and the tin surface will retain its light gray color and the property of holding deposits of electrolytic chromium without peeling.

The second part of this process, that is, the removal of the deposited chromium is accomplished by heating the cathode with the deposit to a point above the transition temperature of beta to gamma tin. This temperature is given as 170° C. by the 18th edition of the "Hand-book of Chemistry and Physics" published by the Chemical Rubber Publishing Company. J. W. Mellor in his book "Modern Inorganic Chemistry" published by Longman's in 1925 lists this change as the transition of tin in the tetragonal system to the rhombic which takes place between 170° and 200° C. It is of course necessary to avoid melting the tin so that at all times the temperature should remain below 231.9° C.

Whatever temperature is used should be maintained for a long enough time to permit substantial conversion of beta to gamma tin to occur. A temperature of 205° C. maintained for about twenty minutes has been found to give good results.

After heating the unstripped cathode should be cooled below the transition point of gamma to beta tin which is 161 degrees centigrade. This may be done by quenching in water or by air cooling as convenient. Stripping may be done at temperatures above 161° C. but cooling is advisable to prevent mechanical damage to the tin as gamma tin is quite brittle and is easily reduced to granular form.

The cooled cathode may now be readily stripped

by first tapping with a light hammer to loosen the chromium and then using a putty knife or other suitable instrument to complete the separation. Generally it may be said that the thicker the deposit of chromium then the more readily it can be stripped, as a very thin deposit exhibits little mechanical strength and offers a very thin edge upon which to pry with a knife or similar instrument.

It will be apparent therefore that the mechanism of weakening the adhesive forces existing between the electro deposited chromium and the underlying tin depends upon the change from beta to gamma tin.

It will also appear that any cathode with a tin surface or an alloy of tin which exhibits this transition in crystal structure will be the equivalent of a tin cathode.

The electrolysis may take place in any suitable apparatus and may proceed along commonly practiced lines. The heating and cooling of the cathode and deposit may likewise be conducted in any suitable manner. The heating, for example, may be done in any oven which may be regulated to preserve a definite range of temperature.

It will be obvious that various changes and modifications may be made in the process above described without departing from the spirit of this invention and limited only by the scope of the following claims.

I claim:

1. A process for preparing chromium metal which consists in electro-depositing chromium metal from aqueous solutions of chromium compounds on an etched-tin cathode, thereafter heating the cathode with the deposit to a temperature above the transition point of beta to gamma tin, cooling said cathode and deposit below the transition point of gamma to beta tin and removing the deposited chromium.

2. A process for preparing chromium which consists in electro-depositing chromium from aqueous solutions of chromium compounds on an etched-tin cathode surface, heating and cooling the deposit and the underlying cathode to loosen the deposit from the said cathode, and subsequently removing the deposit from said cathode.

3. A process for loosening chromium deposits from an etched-tin surface which consists in heating the deposit and tin to a temperature between the transition point of beta to gamma tin and the melting point of tin, then cooling said deposit and tin to a temperature below the transition point of gamma to beta tin.

4. A process for removing chromium deposits from a tin surface which consists in heating the deposit and tin to a temperature above the transition point of beta to gamma tin and below the melting point of tin, cooling said deposit and tin to a temperature below the transition point of gamma to beta tin and removing the deposited chromium.

5. A process for removing chromium deposit from a tin surface which consists in heating the deposit and tin to a temperature above the transition point of beta to gamma tin, and removing the deposit therefrom.

6. The process of removing chromium deposits from an etched-tin cathode which comprises heating the tin and the deposit to a temperature above 170° C. and below 231.9° C., thereafter cooling below 161° C., and then removing the chromium deposit.

7. The process for preparing chromium metal which consists in etching a tin sheet to produce a light gray color and a visible crystal structure, electro-depositing chromium from aqueous solutions of chromium compounds on the etched tin, thereafter heating the tin with the deposit to a temperature above the transition point of beta to gamma tin, and removing the deposited chromium.

JOHN W. BOSS.