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METHOD OF MAKING PULVERULENT CHROMIUM

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This invention relates to making pulverulent chromium; and it comprises electrolyzing a chromium plating solution, usually at a relatively low temperature, with sufficient current density to produce a non-adherent deposit of frangible character, said deposit generally forming curly scales, collecting said deposit, washing and drying it and comminuting; all as more fully hereinafter set forth and as claimed.

Because of the great hardness and strength of the metal chromium, production of fine powdered or granular chromium has offered great technical difficulties. Because of the high heat of oxidation of chromium, it is not practical to reduce it from its oxid by gas currents at a temperature below the melting point, as is done with various other metals. Metallic chromium formed by the aluminothermic process is extremely hard and cannot be economically comminuted or granulated by ordinary grinding machinery to give a powdered product. In granulated chromium so made, the proportion of steel, etc., from the grinding elements is usually great. As a matter of fact, in attempts of this character there was sometimes more wear on the crushing machinery than on the chromium. Electrically deposited chromium produced by the ordinary method is also of extreme hardness and is in thin films cohering to the metal used as the cathode. Sometimes, arborescent deposits, or "trees", occur in ordinary plating methods, but these are also extremely hard and it is not practical to pulverize them to form pure, finely divided chromium.

All the electroplating processes for the deposition of chromium now in use employ a solution of CrO_3 as a bath, this solution containing minor amounts of sulfate and of trivalent chromium. Under proper conditions, very thin, extremely hard deposits can be obtained cohering to the metal plated; a fact which is responsible for the extensive use of chromium plating today. In this plating operation, it is known that better efficiency can be attained at temperatures of 25°C . or below than at higher temperatures. In these plating processes, the current densities are ordinarily such that there is a formation of hydrogen simultaneously with the chromium; but conditions are, of course, so chosen that this formation of gas does not interfere with the plating.

I have found that by using high current densities, densities high enough to cause a copious evolution of hydrogen, in lieu of forming a coherent continuous coating, I produce chromium as non-coherent deposits which can be readily re-

moved. Advantageously, the current density is high enough to produce curly, flaky non-adherent deposits, cracking and breaking away from the cathode. I am of the impression that as the layer on the cathode thickens, the tension of the last formed layer cracks and curls the underlying layers. Chromium formed in this way, after removal from the bath, washing and drying, is of frangible character and can be readily comminuted to a powder as fine as may be desired, without great wear on the crushing apparatus. Whether this frangibility is due to the formation of small independent crystals or is due to inclusion of hydrogen in the metal, I am not aware. It may be due simply to unrelieved tensions in the layer.

The lower the temperature of the bath, the better is the current efficiency and the quicker is the formation of frangible chromium deposits. It is found in practice that there is a lower limit of current density varying with different cathode materials. At less densities, the curling deposit is not formed. Cathodes of aluminum or aluminum alloy are particularly efficient for the present purposes. A certain time is required for the production of detachable deposits. Lead or other unattackable anodes may be employed. Current may be applied until a given bath is exhausted or the bath may be replenished from time to time by additions of CrO_3 in well known manner. Ordinarily, however, I do not use the same bath without interruption for any length of time; this being partly because there is apt to be an accumulation of detached chromium which is slowly dissolved by the bath with consequent waste.

In general the electrolysis is run under conditions so as to form curling, readily frangible flakes carried by the cathode, the cathode being removed before the flakes become thick enough to render crushing too expensive.

In a specific embodiment of the present invention, a bath was made by dissolving commercial CrO_3 to form a solution containing 400 grams per liter. This was acidified by addition of ordinary commercial concentrated sulfuric acid until the total H_2SO_4 content of the bath was 4 grams per liter. In this particular work, an aluminum cathode with about 500 square inches surface area was used, being placed in close proximity to lead anodes. The bath was made cold and kept at a temperature below 25°C . by cooling coils. A current of 2,000 amperes was passed between the electrodes for an hour when a curly non-coherent deposit was formed. At this time,

the cathode was removed from the bath and adherent chromium removed. Detached chromium occurring in the bath was also collected. The chromium was washed and dried and then crushed to a powder. In this particular work a ball mill was used.

Chromium made in the described manner is frangible enough to permit the use of any convenient crushing device.

The limiting current density varies somewhat with the cathode material and with the temperature, but using aluminum cathodes and a temperature below 25° C., it is of the order of 2 amperes per square inch.

The temperature of the bath is not specially important except as it controls efficiency. Good flaked deposits can be obtained at temperatures as high as 48° C. on nickel, aluminum and aluminum alloys.

Other chromium plating baths than solutions of CrO_3 can be used; but the latter are the best at present known.

As a rule, the flaked chromium is washed and dried prior to comminuting, but it is sometimes convenient to wet grind the flakes to form a paste or pulp and afterwards dry this to produce the desired pulverulent chromium.

The present method may be applied to making granular chromium or pulverulent chromium of any mesh desired. The flake chromium is sometimes utilized as such.

The product produced according to my invention has many uses, for example, in the manufacture of inks, paints, coatings, alloys, etc.

It is to be understood that the invention is not limited to any specific set of conditions described above, but may be more or less materially modified without departure from the spirit and scope of the same as called for in the following claims.

What I claim is:

1. A process of producing metallic chromium as a fine powder which comprises electrolyzing

a chromium plating bath containing a high concentration of CrO_3 with a cathode made of a metal not attacked by the bath, maintaining a high current density on said cathode with temperature and other conditions producing a copious evolution of hydrogen gas and a deposition of chromium upon the cathode in non-adherent brittle flakes, detaching said flakes from the cathode and grinding the flakes to a powder.

2. The process of claim 1 wherein the electro-deposition is effected on an aluminum cathode.

3. The process of claim 1 wherein a current density above 2 amperes per square inch of cathode is employed.

4. In the process of claim 1, maintenance of the plating bath at a temperature not above 25° C.

5. In the production of pulverulent or granular chromium, the process which comprises electrolyzing a bath of CrO_3 at a low temperature and at a high cathode current density giving metallic chromium in detachable flakes at the cathode with a copious evolution of hydrogen gas, washing and drying the detached flakes and comminuting to the fineness desired.

6. In the production of readily crushable chromium, the process which comprises electrolyzing a bath of CrO_3 with an aluminum cathode at a temperature not above 48° C. and at a sufficiently high cathode current density to give metallic chromium in detachable flakes with a copious evolution of hydrogen.

7. In the production of readily crushable chromium, the process which comprises electrolyzing a bath of CrO_3 with an aluminum cathode at a temperature not above 48° C. and at a sufficiently high cathode current density to give metallic chromium in detachable flakes with a copious evolution of hydrogen, and washing and drying the detached flakes.

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