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**Index at acceptance— 130 F [ XXXIII (7) ]  
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International Classification— C 23 f 3/02.**

**" PROCESS FOR CHROMATE TREATMENT OF ZINC  
AND DIE CAST ZINC ALLOY FOR CORROSION PROTECTION".**

**COUNCIL OF SCIENTIFIC & 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.**

**PRICE : TWO RUPEES**

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This invention relates to improvements in or relating to chromate treatment of zinc and die cast zinc alloy.

Hitherto it has been proposed that the chromate treatment bath is an acidic aqueous solution of soluble hexavalent chromium compound + one or more activators. The typical formulation for chromating zinc available in literature is one of 180- 200 g.p.l. sodium or potassium dichromate acidified with 6- 6.5 cc/ litre of concentrated sulphuric acid with activators such as fluoride, nitrate borate, acetate etc.

This is open to <sup>the</sup> objection that ( a ) olive green chromate coating having the highest corrosion resistance is not produced in these baths on zinc or zinc cast alloy and ( b ) iridescent coatings having pleasing appearance are not obtained at die- cast zinc alloy.

The object of the present invention is to obviate these disadvantages by producing an olive green coating with the iridescent appearance.

To these ends the invention broadly consists in formulating a chromate treatment bath containing 80-200 grams of potassium or sodium dichromate 5-25 grams of sulphuric acid, 0.5-5 grams of sodium fluoride and 5-25 grams of sodium nitrate and 0.1 to 1 gram of wetting agent per litre of solution to get an iridescent yellow or iridescent olive green colour.

The following typical examples are given to illustrate the invention.

Example I:

1" x 1" zinc plate with stem is taken and pickled in 20% chromic acid at 80°C for 5 minutes to remove oxide scale on the surface. After pickling, the specimen is washed in tap water and then dipped in the solution containing 100 g/l of potassium dichromate, 10 ml/l of sulphuric acid (s.p.gr. 1.84) 1 g/l of lauryl sulphate and 0.5 grams/litre of sodium fluoride for 10 seconds to get an olive green iridescent chromate coating on the surface.

Example II:

Maxak alloy (diecast-zinc alloy) specimen is pickled in 20% chromic acid for about 10 minutes at 80°C to remove the scale on the surface. After pickling, the specimen is washed in tap water and then dipped in the solution containing 100 g/l of potassium dichromate, 10 millilitres per litre of sulphuric acid (s.p.gr. 1.84), 1 g/l of CTAB agent and 0.5 grams/litre of sodium fluoride for 1 minute to get an olive green iridescent chromate coating on the surface.

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Example III

Mazak alloy component is cleaned in alkaline cleaner containing 6 g/l each of sodium hydroxide and sodium carbonate at 90-95°C for 30 seconds, rinsed in tap water, then dipped in solution containing 120 g/l of sodium-dichromate, 12 g/l of sulphuric acid, 0.5 g/l of sodium fluoride and 5 g/l of sodium nitrate for 20-30 seconds to get an iridescent chromate coating on the surface.

Comparative Corrosion resistance is given in Table 1.

Table 1.

Passivating treatment	Hours of resistance to salt spray	
	Zinc	5% NaCl Die-cast zinc
1 None	26	10
2 200 g/l of dichromate + 6 cc/l. conc. H <sub>2</sub> SO <sub>4</sub>	95	49
3 Proprietary	35	33
4 Patented formulation	125	49

The colour of chromated surface thus obtained is compared with compositions mentioned in literature and proprietary baths in Table 2.

Table 2:

Raw material cost and area treated:

Diecast zinc surface was treated from 20 seconds upto 60 seconds and the bath was not replenished from any of its constituent. The cost figures are about the same for the two baths.

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Table-2

Colour of Chromated Surface:

<u>Passivating composition</u>	<u>Zinc</u>	<u>Die Cast Zinc</u>
1. 180-200 gpl. dichromate & 6 cc/l conc., sulphuric acid	Golden yellow	Dark grey or brown
2. (1) Containing 2.5 gpl sodium nitrate	Interference film.	Dirty yellow
3. (1) Containing 1 gpl sodium fluoride.	Golden yellow	Dark grey.
4. Proprietary	Bluish grey	Bluish grey
5. Patented	Iridescent yellow	Iridescent yellow.

Following are among the main advantages of the inventions:-

1. An olive green coating with iridescent appearance is obtained on both zinc and die-cast zinc alloy unlike the compositions mentioned in literature.
2. The cost of this treatment is similar to the cost of treatments mentioned in literature (Table I).

Dated this 9th day of June, 1975.

  
ASST. PATENTS OFFICER,  
COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

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**THE PATENTS ACT. 1970**

**COMPLETE SPECIFICATION**

( Section—10 )

1. PROCESS FOR COALING ZINC AND DIE CAST ZINC ALLOY FOR CORROSION PROTECTION
2. 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 particularly describes and ascertains the nature of this invention and the manner in which it is to be performed:-

This is an invention by Kumattithidal Santhanam RAJAGOPALIAN, Palaniyappa ANNAMALAI, Mrs VENU Subramanyan, Scientists , Chakrabarthi RAJAGOPAL , Senior Scientific Assistant and Mrs Vijayalakshmi Ramakrishnan , Senior Laboratory Assistant- all from Central Electrochemical Research Institute, Karaikudi, Tamil Nadu- India- and Indian Citizens.

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This invention relates to process for <sup>c</sup>chromate treat- *Coating*  
ment of zinc and die-cast zinc alloy and relates to <sup>in</sup>Metal  
f/finishing industry.

Hitherto it has been proposed that chromate treatment bath is an acidic aqueous solution of soluble hexavalent chromium compound plus one or more activators.

This is open to the objection that (a) olive green chromate coating having the good corrosion resistance is not produced in these baths on zinc or zinc die-cast alloy and (b) iridescent coatings having pleasing appearance are not obtained on die-cast zinc alloy.

The object of this invention is to obviate this disadvantage by producing an olive green or yellow coating with the iridescent appearance having good corrosion resistance.

The main finding is the formulation of a chromate treatment bath containing 80-200 grams of potassium or sodium dichromate, 5-25 grams of sulphuric acid 0.5-5 grams of sodium fluoride and 5-25 grams of sodium nitrate and 0.1 to 1 gram of wetting agent (sodium lauryl sul. hate ~~etc. P.A.B.~~) per litre of solution to get an iridescent yellow or iridescent olive green colour with good corrosion resistance properties.

The formulation gives an iridescent olive green or yellow coating having good corrosion resistance and pleasing appearance.

The present invention consists of a process which comprises of the incorporation of a wetting agent and an activator in the chromate passivating solution, which makes it possible to get a decorative and corrosion resistance coating.

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The following typical examples are given to illustrate the invention.

Example I

1" x 1" zinc plate with stem is taken and pickled in 20% chromic acid at 80°C for 5 minutes to remove oxide scale on the surface. After pickling, the specimen is washed in tap water and then dipped in the solution containing 100 g/l of potassium dichromate 10 ml/l of sulphuric acid (Sp.gr 1.84) 1 g/l of sodium lauryl sulphate and 0.5 grams/litre of sodium fluoride for 10 seconds to get an iridescent chromate coating on the surface.

Example II

Masak alloy (die-cast zinc alloy) specimen is pickled in 20% chromic acid for about 10 minutes at 80°C to remove the scale on the surface. After pickling, the specimen is washed in tap water and then dipped in the solution containing 100 gpl. of potassium dichromate, 10 ml per litre of sulphuric acid (Sp.gr 1.84), 1 g/l of CTAB agent and 0.5 grams/litre of sodium fluoride for 1 minute to get an olive green iridescent chromate coating on the surface.

Example III

Masak alloy component is cleaned in alkaline cleaner containing 6 g/l each of sodium hydroxide and sodium carbonate at 90-95°C for 30 seconds, rinsed in tap water, then dipped in solution containing 120 g/l of sodium dichromate, 12 g/l of sulphuric acid 0.5 g/l of sodium fluoride and



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5 g/l sodium nitrate for 20-30 seconds to get an iridescent yellow chromate coating on the surface.

- (a) The corrosion resistance of chromate specimens passivated in the chromate solution, developed by us is given below, in comparison with unchromated zinc specimens. Comparative corrosion resistance is given in table 1.

Passivating treatment	Hours of resistance to Salt Spray (5% NaCl)	
	Zinc	Die-cast zinc alloy
1. None	26	10
2. 200 gpl of dichromate + 6 ml/l Conc. $H_2SO_4$	95	49
3. "Ash chrome -Z" (chromate solution manufactured and marketed by M/s Ashok Industries, Bombay, the composition of which is not known).	35	33
4. (80-200 g.p.l. dichromate (potassium or sodium)  5-25 gms sulphuric acid 0.5 to 5 gms sodium fluoride 5-25 gms. sodium nitrate 0.1 to 1 gm wetting agent (CTAB) (all in gms/litre)	125	49

- (b) The colour of chromated surface thus obtained is compared with compositions mentioned in literature and proprietary baths in Table 2.

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TABLE 2

Sl.No.	Passivating composition	Zinc	Die-cast zinc alloy.
1.	180-200 g.p.l. dichromate + 6 ml/l Conc., H <sub>2</sub> SO <sub>4</sub>	golden yellow	Dark grey or brown.
2.	(1) containing 2.5 g.p.l. sodium nitrate.	interference film.	Dirty grey
3.	(1) containing 1 gpl sodium fluoride.	golden yellow.	Dark grey
4.	Ash chrome -Z (chromate solution manufactured and marketed by M/s Ashok Industries, Bombay the composition of which is not known).	bluish grey.	bluish grey
5.	(80-200 g.p.l. dichromate (potassium or sodium) 5-25 gms sulphuric acid 0.5 to 5 gms. sodium fluoride 5-25 gms. sodium nitrate 0.1 to 1 gm wetting agent (CTAB) (all in gms/litre)	olive green	iridescent olive green/yellow.

An olive green/yellow coating with iridescent appearance is obtained on both zinc and die-cast zinc alloy unlike the compositions mentioned in literature.

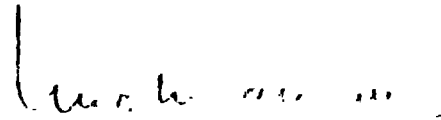
The invention consists in formulating a chromate passivating composition for zinc and die-cast zinc alloys, which produces an iridescent coating having good corrosion resistance.

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We claim:-

A process for coating zinc and die - cast zinc alloys for corrosion protection wherein the metal piece is dipped in a chromate bath, characterised in that the bath comprises 80-200 gms./litre of potassium or sodium dichromate 5-25 gms./litre of sulphuric acid, 0.5-5 gms./litre of sodium fluoride, 5-25 gms./litre of sodium nitrate and 0.1-1 gms./litre of a wetting agent such as sodium lauryl sulphate.

Dated this 30th day of December, 1975.



( I.M.S. MAMAK )  
SCIENTIST 'E' (PATENTS)  
COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH.