



Bright zinc electrodeposition and study of influence of synergistic interaction of additives on coating properties



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ABSTRACT

The nanocrystalline, bright zinc coating was produced using additives, cetyltrimethylammonium bromide (CTAB) and condensation product of valine and veratraldehyde (VV). The synergistic influence of VV + CTAB and simultaneously effect of VV content in bath on surface morphology, texture and grain size of zinc electrodeposits were systematically analyzed. In addition, effect of additives on efficiency, throwing power and deposit properties like microhardness, reflectance and corrosion resistance was investigated. The study reveals that morphological and structural changes observed in presence of additives exhibits synergistic influence on improving corrosion resistance and hardness of the deposit.

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Introduction

The zinc electrodeposition has been widely used to generate corrosion resistant coating on steel due to its sacrificial nature [1,2]. In majority of industrial applications nanocrystalline, bright zinc coating is preferred than dull coating, as it exhibits relatively better corrosion resistance and other functional properties [3–5]. Hence, the development of nanocrystalline, bright deposit over spectrum of current density from a suitable electrolyte is always in demand. The nanocrystalline, bright zinc coating is generally obtained from electrolyte solutions containing certain special compounds in small amount called additives. Many such additives like aromatic and aliphatic aldehydes, ketones, carboxylic acid, polymers, amines and condensation products of some simple compounds are reported in the literature [6–9].

Usually, acid chloride [10], sulphate [11], mixed sulphate chloride [12] and alkaline [13] types of bath are frequently employed in zinc electroplating. Out of these, sulphate bath possesses relatively better plating properties due to nonpolluting nature of its constituents [11,14]. The good additives, developed in sulphate bath generates leveled, nanocrystalline, bright deposit over wide

current density range and improves mechanical and physical properties of deposit [15–18].

In addition use of suitable combination of two or more additives results in further improvement of deposit properties and performance of plating bath. Moron et al. investigated zinc coatings obtained in presence of brightener-PEG200 (polyethyleneglycol MW200) associate showed lower corrosion current density than the coatings formed in the absence of additives and presence of PEG200 alone. Also zinc coating morphology changes from hexagonal crystals with a (1 0 1) preferred orientation in the absence of additives, to flakes grouped in hemispherical clusters with (0 0 2) (1 0 0) (1 0 1) crystallographic orientations when brightener-PEG200 associate is present in the bath [15]. Hsieh et al. reported the synergistic interaction among additives NP16 (polyoxyethylene nonyl phenyl ether), OCBA (*o*-chloro benzyl aldehyde) and S40 (polyoxyethylene lauryl amine), effects on smoothening zinc deposit and increasing current efficiency at high current density [17]. Loto developed bright, corrosion resistant zinc electrodeposit using synergistic combination of pineapple and sugar extract [19]. In another work Loto also reported the formation of leveled, bright and corrosion resistant zinc electrodeposit with good throwing power in presence of additives dextrin and thiourea [20]. The existing reports reveals that zinc plating baths associated with two or more additives generating bright deposit with more pronounced plating bath and deposit properties. But the study regarding the synergistic effect of additives on

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morphology, orientation and properties of deposit is limited. Hence the detail research is required to examine the influence of additive combination and their concentration on properties of deposit.

In the present work, new bright zinc electroplating bath (using additives, CTAB and VV) capable of generating bright deposit over a wide range of current density is employed. The effect of additives, CTAB and VV individually and their combination on current efficiency, throwing power of bath and coating properties are studied. In addition, efforts have been made to investigate the influence of VV concentration in optimum bath on surface morphology, orientation, microhardness and corrosion resistance of bright zinc deposit.

Experimental

The bath solution was prepared by dissolving analytical grades (SD Fine Chemicals, Mumbai, India) of $160 \text{ g L}^{-1} \text{ ZnSO}_4 \cdot \text{H}_2\text{O}$, $40 \text{ g L}^{-1} \text{ Na}_2\text{SO}_4$, $24 \text{ g L}^{-1} \text{ H}_3\text{BO}_3$, and $2 \text{ g L}^{-1} \text{ CTAB}$ (cetyltrimethylammonium bromide) in Millipore (Elix France) water. The pH of bath was adjusted to 2.5 using 10% H_2SO_4 and NaHCO_3 solutions. The bath constituents and operating conditions are optimized by Hull cell studies. The 99.9% pure zinc anode of $5 \times 6 \text{ cm}^2$ area was used as anode and its surface was activated each time by dipping in 10% HCl for few seconds followed by water wash. The mild steel plate of Hull cell dimension ($4 \times 6 \text{ cm}^2$ for other studies) was used as cathode. The surface of mild steel plates were polished mechanically with 800 grit waterproof abrasive paper, degreased by vapors of trichloroethylene in degreaser plant followed by 30 s immersion in 10% HCl to remove dust and rust. The plates were then washed in running water, and transferred to electroplating bath. After 10 min plating at 1 A, plates were given bright dip in 1% HNO_3 for 2 s followed by water wash and air drying. The bath temperature was maintained at $303 \pm 3 \text{ K}$ by means of a thermostat.

The VV was prepared as follows: 0.50 g of veratraldehyde dissolved in 5 mL of acetic acid solution was heated to activate aldehyde. 0.35 g of valine dissolved in 5 mL water was added drop wise to the solution and refluxed for 3 h at 70°C . The yellow coloured solution was obtained and the completion of the reaction was tested by TLC. Above solution is diluted up to 100 mL and known quantity is used for further experiment.

Current efficiency (CE) was measured using rectangular cell of 0.5 L capacity with dimension $20 \times 5 \times 6 \text{ cm}^3$. The cathode of exposed area $4 \times 4 \text{ cm}^2$ was placed between two anodes at equal distance in rectangular cell. Plating was carried out for 10 min and the weight of deposit was measured. This weight was compared with theoretical weight from Faraday's law and the current efficiency was calculated.

Throwing power was measured in absence and presence of additives using Haring and Blum cell and calculated using Field's formula

$$\text{throwing power (\%)} = \frac{K - C}{K + C - 2} \times 100 \quad (1)$$

where C is metal distribution ratio between the near and far cathode and K is ratio between the respective distance of the far and near cathode from anode.

The cathodic polarization studies were carried out by using three compartment cell. The mild steel of $1 \times 1 \text{ cm}^2$ and platinum wire were used as working and auxiliary electrode, respectively. The potential was measured with reference to saturated calomel electrode.

Morphology of the deposits was analyzed by scanning electron microscope (FESEM, LE01530-VP) images. Also grain size and crystal orientations of the deposits were examined by X-ray diffraction (XRD) studies using PANalytical X'pert Pro powder

diffractometer. The average grain size of deposits was calculated using Debye Scherer's equation $D = k\lambda/\beta\cos\theta$. Where, $k = 0.9$, β is full width at half maximum, θ is reflectance angle and $\lambda = 1.546 \text{ \AA}$ wave length of radiations used [21].

The preferred orientations of deposits were determined by Muresan's method by calculating the texture coefficient (T_c) using equation [22–24].

$$T_c(hkl) = \frac{I(hkl)}{\sum I(hkl)} \times \frac{\sum I_o(hkl)}{I_o(hkl)} \times 100 \quad (2)$$

where $I(hkl)$ is the peak intensity of zinc electrodeposits and $\sum I$ is the sum of the intensities of independent peaks. The index 0 refers to the intensities for the standard zinc sample (JCPDS card number: 00-004-0831).

The Vickers microhardness of the deposits was determined by indentation technique with a weight of 25 g and 50 g for 5 s using Clemex microhardness tester, made in Japan. Four spots at different locations on each zinc coated surface were used for microhardness measurement and average of these gave microhardness value.

Zinc coating was obtained from the optimized bath solution in the absence and presence of CTAB, VV and CTAB + VV. Beside that coatings were also obtained in presence of different concentration of VV (16 to 72 mL L^{-1}) at constant amount of CTAB. These coatings were subjected to reflectivity measurement using gloss meter (NOVO gloss meter) referenced against a vacuum coated silver mirror. The reflectivity of mirror was set at 100% at an angle of 60° . The measurements were carried out at different points on the coating surface and the average value was reported [25].

The standard bending test was used to measure adherence and ductility of zinc deposit. The $2 \times 5 \text{ cm}^2$ zinc coated steel strips were bent forward and backward through 180° , the crack developed was taken as the measure of adherence and ductility of deposit.

The corrosion behavior of $4 \times 4 \text{ cm}^2$ coated samples in 3.5% NaCl solution was investigated by weight loss method. After specified hours of immersion the mass loss incurred by them with respect to time was determined.

The corrosion behavior of coatings was also analyzed by electrochemical methods (anodic polarization, Tafel and impedance) in 3.5% NaCl solution using conventional three electrode cell and CHI 660C, electrochemical work station (USA). The coated steel samples with $1 \times 1 \text{ cm}^2$ exposed surface area, saturated calomel electrode and Pt wire were employed as working, reference and counter electrodes, respectively.

Results and discussion

Using additives VV and CTAB bright zinc plating bath was optimized by Hull cell experiments. During Hull cell studies concentration of bath constituent and operating conditions were optimized. The concentration of one constituent was varied in constant increments while keeping other constituents at their selected concentration. The particular concentration at which bright, uniform deposit produced over wide range of current density was fixed as optimum concentration. The procedure was repeated for all bath constituents and operating conditions. The optimum bath composition and operating conditions selected are given in Table 1.

In optimized bath, CTAB serve as a surfactant and VV as a brightener. A mixture of these two additives in the bath generates smooth, uniform, bright deposit over broad range of current density compare to presence of either CTAB or VV. To know the influence of individual additives as well as their combination on bath characteristics and deposit properties a detail study was undertaken. Also the brightening agent VV in optimized bath plays an important role on improving performance of bath solution and

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