

A study on brightening property of newly synthesized compound in electroplating of zinc-nickel alloy

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The electrodeposition of zinc-nickel (Zn-Ni) alloy from chloride bath was carried out in presence of condensation product formed between thiamine hydrochloride (THC) and furfural (FFL). The bath constituents were optimized through Hull cell experiments. Operating parameters such as pH, temperature and current density were also optimized. Current efficiency and throwing power were measured. Polarization study revealed shift of potential towards negative direction in the presence of addition agents. Corrosion resistance tests revealed good protection of base metal by Zn-Ni alloy deposit. SEMs showed fine-grained deposit in the presence of condensation product. FTIR spectrum of the scrapped deposit showed the inclusion of condensation product in the deposit during plating. The consumption of brightener in the lab scale was 5 mL⁻¹ for 1000 amp-h.

Keywords: Chloride bath, Electroplating of zinc-nickel, Furfural, Hull cell studies, Thiamine hydrochloride

The corrosion resistance of conventional Zn coating is not sufficient nowadays because of the permanent requirement of industry (especially automotive) to reduce coating thickness and to increase the corrosion resistance at the same time^{1,2}. Therefore, the conventional Zn coatings are being replaced by Zn alloys³. The corrosion resistance of electrodeposited Zn-Ni alloy coatings within a certain composition range (9-15 wt%) can be significantly higher (five to six times) than that of pure Zn^{4,5}. Electrodeposited Zn-Ni alloys and their coatings are the perfect coatings instead of chromium⁶. The deposition of Zn-Ni have been obtained from various types of baths like chloride, sulphate, sulphate-chloride, and alkaline⁷⁻⁹. Among these chloride baths are preferred because of their higher conductivity, low cost and non-polluting nature¹⁰. The use of additives in electrolytic baths is very important since they influence the growth and the structure of the deposits¹¹. Typically, additives are added to the electrolytic bath at concentrations in the order of parts per million; their presence in the baths promotes the formation of soft and shiny coatings¹². In recent years condensation product of aldehyde and amine have been extensively used as additives in the electrodeposition of Zn and Zn alloys. They are good brighteners than aldehyde or amine alone in the bath solution¹³⁻¹⁷.

In the present study efforts have been made to study the effects of newly synthesized additive on Zn-Ni alloy plating from acid chloride bath. Results on current efficiency, throwing power, cathode polarization and corrosion resistance have also been reported.

Experimental Procedure

The chemicals used were of AR grade and easily soluble in water. For the preparation of solutions distilled water was used. The standard Hull cell of 267 mL capacity was used to optimize the bath constituents with an anode dimension of 6 × 6.5 cm and cathode dimension of 10 × 7.5 cm. The Hull cell experiments with the bath solutions were carried out without agitation. The pH of the bath solution was adjusted with 10% sulphuric acid or sodium bicarbonate solution. Zn plate of 99.99% purity was used as anode. The anode was activated each time by immersing in 10% HCl followed by washing with water. Mild steel plates (AISI-1079) of standard Hull cell size were mechanically polished to obtain a smooth surface and degreased by dipping in boiling trichloroethylene. The scales and dust on the steel plates were removed by dipping in 10% HCl solution and were subjected to electrocleaning process. Then these steel plates were washed with water and used

for experiments as such. After the experiment the plates were subjected to bright dip in 1% nitric acid for 2-3 s followed by water wash. The nature and appearance of the alloy deposit was carefully studied and recorded through the Hull cell codes Fig. 1(a).

All the experiments were conducted at 303 ± 1 K. The condensation product was synthesized¹⁸⁻²⁰ by refluxing equimolar amounts of THC (2.5 g) and FFL (0.61 mL) in ethanol (50 mL), for 3 h at 343 K. The resulting product was diluted to 250 mL with distilled water. A known amount of this solution was added to the electroplating bath and the solution was stirred for 30 min before conducting the experiment.

Ni content in the plating layer was analyzed gravimetrically by the procedure given elsewhere²¹.

The deposits were obtained at constant current density from the optimized solution taken in a rectangular methacrylate cell of 2.5 L capacity. Polished, degreased and electrocleaned cathodes of 3×4 cm were used for plating experiments. These plated steel cathodes were used to test different metallurgical properties. Experiments were done in triplicate. Standard experimental procedures²² were adopted for the measurement of metallurgical properties of the deposit such as ductility, hardness, adherence etc. In all the above studies the average thickness of the deposit was 20 μ m. The coating thickness was measured by using β -ray back scattering gauge (Permascope ESD9, West Gut-ESD9 KB4, 220X50-60 Hz, German) and BNF jet methods.

For corrosion resistance test, the coated steel plates of 3×4 cm area were given bright dip followed by passivation in a solution containing 200 g L^{-1} sodium dichromate and 2 mL L^{-1} of sulphuric acid at 303 K for 5 s. These passivated samples were dried for 24 h in a clean and dry atmosphere and then subjected to neutral salt spray test in accordance with ASTM standard method B-117 using 5% neutral sodium chloride solution at 303 K.

Polarization studies were carried out by using three compartment cell. The exposed area of Zn anode was 2 cm^2 . Mild steel was used as cathode with an exposed area of 2 cm^2 . The cathode potential was recorded, galvanostatically, with respect to saturated calomel electrode at different current densities.

Haring and Blum cell was used to measure throwing power of the bath solution and a rectangular methacrylate cell was used to measure current efficiency. For throwing power measurement the

current distribution ratio between anode and cathodes was 1:5.

The more useful method for measuring microhardness involved making an indentation with an indenter of specified geometry under specified load. The length of indentation was measured and microhardness calculated (Vicker hardness tester, British make). The value was expressed in Vickers hardness number (VHN). Alloy thickness of 20 μ m and load of 50 g was employed.

FTIR spectrum of the scrapped Zn-Ni deposit was recorded using Perkin-Elmer 1760 instrument. SEM photomicrographs were taken (JEOL-JSM-35 LF) to know the surface morphology of deposit in the presence of addition agent.

To know the amount of addition agents consumed in the bath, 2.5 L of bath solution was taken and plating was carried out at different current densities. The total number of coulombs passed to the bath solution was recorded at the time when the bath just started to give semibright deposit. The bath solution after use was subjected to Hull cell test by adding different amounts of condensation product. The concentration of the condensation product, at which once again bright deposit was obtained, was recorded.

Results and Discussion

Effect of condensation product

The Hull cell experiments using basic bath solution (Table 1) gave coarse dull deposit in the current density range between 1 - 4 Adm^{-2} at 1A cell current. To improve the nature of the deposit condensation product formed between THC and FFL was added to the bath solution. The electroactive functional groups of the condensation product form complexes with Zn and Ni metal ions in the solution and control the rate of metal deposition. The deposition potential for metal ions in presence of additive shifted to more negative direction and led to a regular and uniform crystallization.

At lower concentration ($< 20 \text{ mL L}^{-1}$) of the condensation product, the deposit was semibright in the current density range of 1.5-3 Adm^{-2} . At low

Table 1—Basic bath composition and operating conditions

Bath composition	Quantity (g L^{-1})	Operating conditions
ZnCl ₂	25	Anode: Zinc metal (99.99%)
NiCl ₂	3	Cathode: Mild steel
NH ₄ Cl	200	Temperature: 293-303 K
H ₃ BO ₃	20	Cell current: 1A

current density region dull and at high current density region burnt deposits were obtained. With increase in the concentration, nature of the deposit was improved and at a concentration of 20 mL^{-1} of condensation product, the Hull cell panels were bright in the current density of $0.5\text{--}4 \text{ Adm}^{-2}$. With further increase in the concentration of condensation product, the nature of deposit became brittle at high current density region. Therefore, on the basis of above observations the concentration of condensation product was kept at 20 mL^{-1} as optimum. The Hull cell panels exhibiting the effect of condensation product are shown in the Fig. 1(b) and the relation between concentration of condensation product and Ni content is illustrated in Fig. 2 (a).

Effect of zinc chloride

There is close relation between the concentration of zinc chloride and appearance of plating layer in Hull cell experiment. Veeraraghavan *et al.*²³ reported that in order to get high corrosion resistance and bright Zn deposit it is essential to strike a balance between amount of Zn content in the bath solution. To find out the effect of Zn metal ion concentration on the nature of deposit, zinc chloride concentration was varied from 10 to 50 gL^{-1} . At concentrations $< 30 \text{ gL}^{-1}$, bright deposit was observed in the current density range of $2\text{--}4 \text{ Adm}^{-2}$. At low current density region, uncoated and at high current density region burnt deposits were obtained. With increase in the concentration of zinc chloride, the brightness range was extended to high

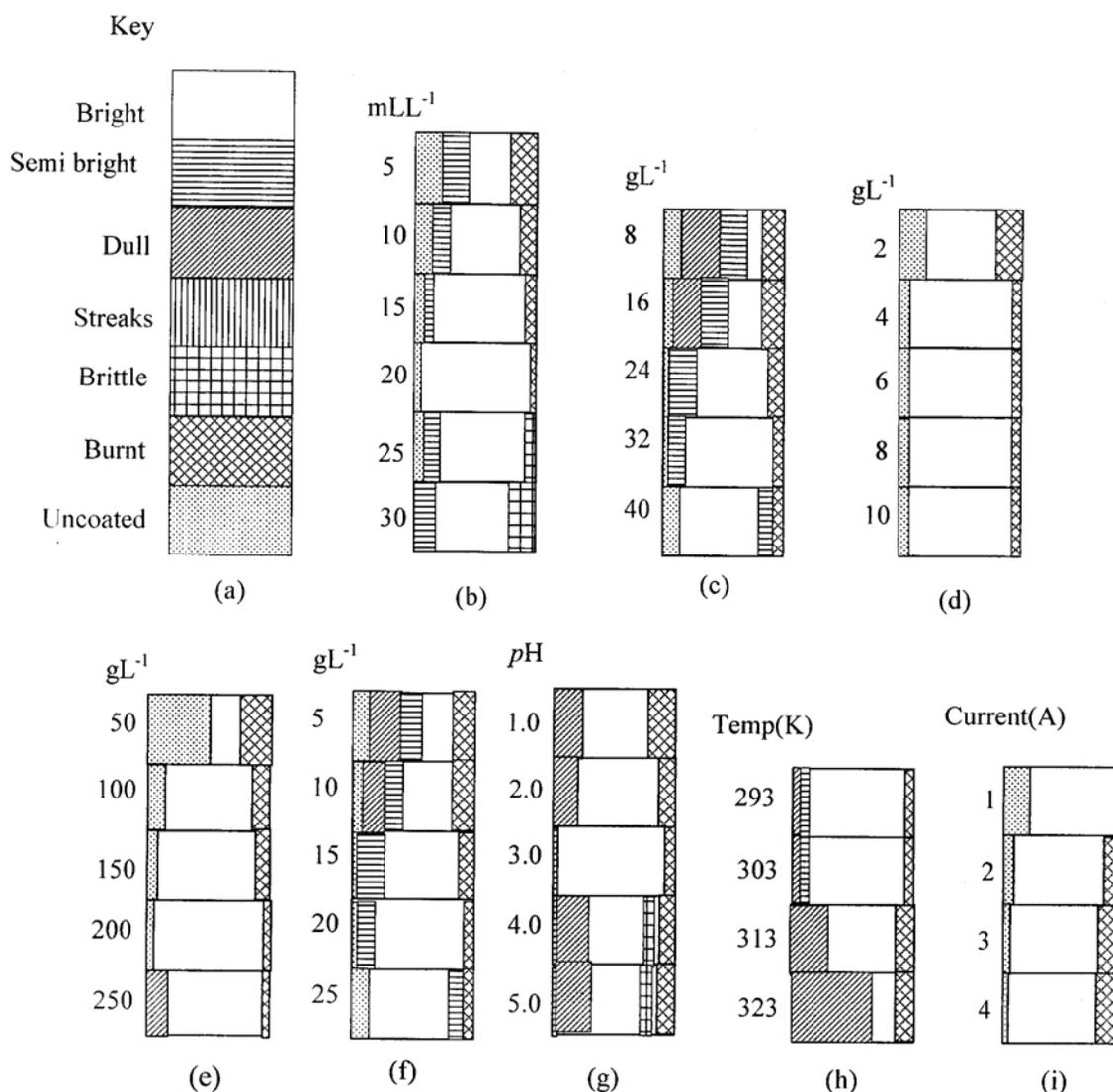


Fig. 1—Hull cell figures: (a) Key, Effect of: (b) condensation product, (c) ZnCl_2 , (d) NiCl_2 , (e) NH_4Cl , (f) H_3BO_3 , (g) pH, (h) temperature, and (i) cell current.

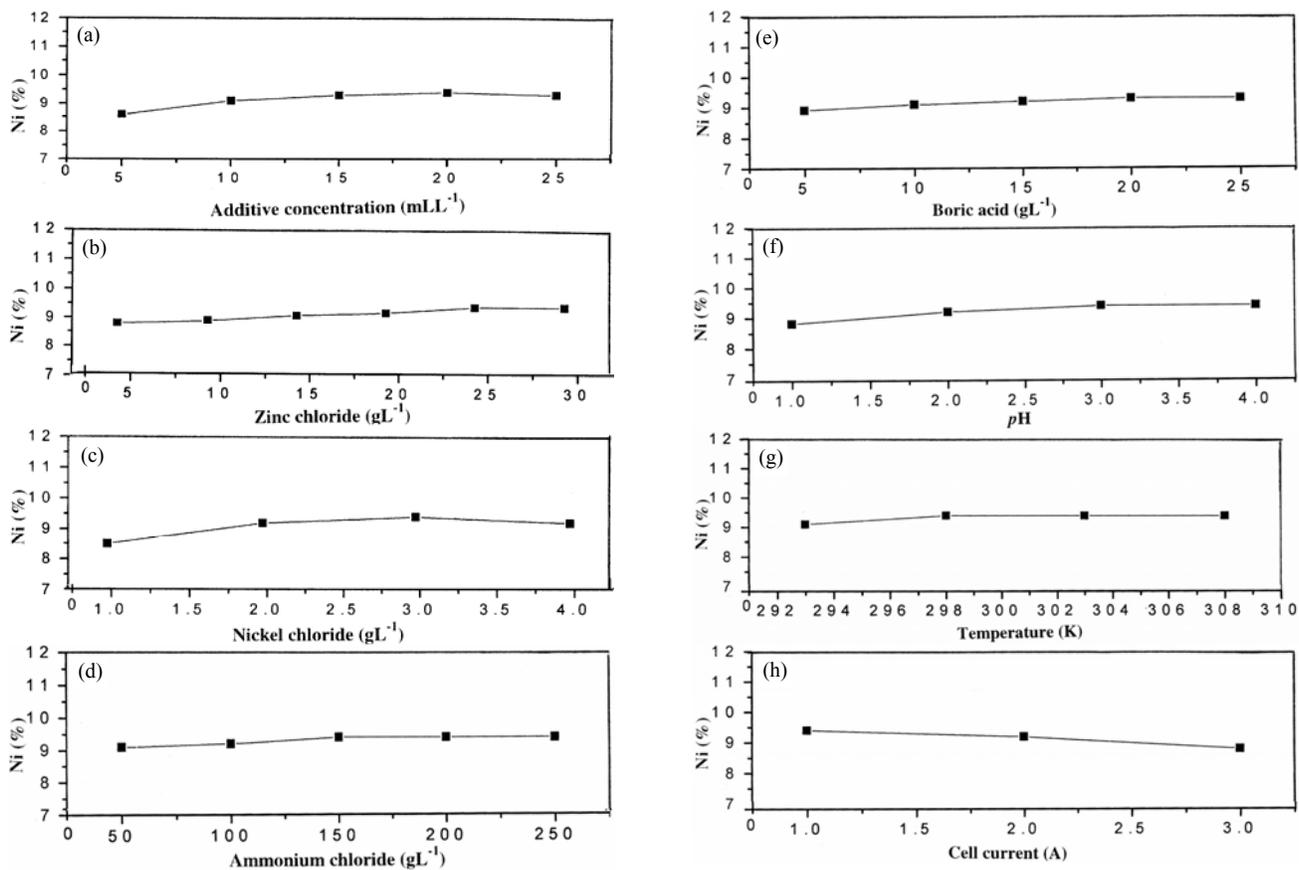


Fig. 2—Effect of: (a) additive, (b) zinc chloride, (c) nickel chloride (d) ammonium chloride, (e) boric acid, (f) pH, (g) temperature and (h) cell current, on Ni content of the plating layer

and low current density regions. At a concentration of 32 gL⁻¹, satisfactory bright deposit was obtained in the current density range of 0.5-4 Adm⁻² at 1A cell current. With further increase in the concentration of zinc chloride no improvement in the nature of the deposit was observed. The effect of zinc chloride on nature of deposit is shown in Fig. 1(c) and also the relation between zinc chloride and Ni content is illustrated in Fig. 2 (b).

Effect of nickel chloride

At a particular concentration of Ni ions in the solution anode was properly beneficial to dissolution, and cathode polarization increased, so a fine crystallized layer was obtained. Roev *et al.*²⁴ showed that 3 gL⁻¹ of Ni gave fine grained deposit in alkaline Zn-Ni alloy plating bath, further increase in the concentration of Ni produce black powdered deposit. So it is necessary to optimize the concentration of Ni in the bath solution. Nickel chloride concentration was varied from 1-5 gL⁻¹. At concentrations < 4 gL⁻¹, the bright deposit was observed in the current density range between 2-4 Adm⁻². At low current density

region uncoated and at high current density region burnt deposits were obtained. With increase in the concentration of nickel chloride, the brightness range was extended to high and low current density regions. At a concentration of 4 gL⁻¹, satisfactory bright deposit was obtained in the current density range of 0.5-4 Adm⁻² at 1A cell current. With further increase in the concentration of nickel chloride no improvement in the nature of deposit was observed. Hence, the concentration of nickel chloride was fixed at 4 gL⁻¹ as optimum. The effect of nickel chloride on deposit nature is shown in Fig. 1(d) and also the relation between nickel chloride and Ni content is illustrated in Fig. 2 (c).

Effect of ammonium chloride

There was a co-relation between the ammonium chloride content and appearance of plating layer in Hull cell experiment, as ammonium chloride was lower than 150 gL⁻¹, bright deposit appeared on the side of low current density region, at ammonium chloride content higher than 200 gL⁻¹, ammonium chloride could easily crystallized. According to the

Hull cell experiment, the appropriate amount of ammonium chloride in plating solution ranged from 200-250 gL⁻¹, and the optimum value was 220 gL⁻¹. The effect of ammonium chloride on nature of deposit is shown in Fig. 1(e) and the relation between ammonium chloride and Ni content is illustrated in Fig. 2 (d).

Effect of boric acid

The role of boric acid has been of great interest in the electrodeposition of Zn-Ni alloy²⁵. It is now believed that boric acid either complexes with Ni²⁺, acting as homogeneous catalyst, or adsorbs on the electrode surface, has significant role on morphology and compositional characteristics. The presence of boric acid results in an increase of current efficiency of deposition processes, amount of Zn in the deposited alloy, and the nucleation density of the deposit. These effects have been attributed to the adsorptive interaction of boric acid at the electrode surface. Also, boric acid acts as buffer to maintain the pH of the electrolyte bath^{26,27}.

To determine the optimum concentration of boric acid, its concentration was varied from 10-30 gL⁻¹ keeping the concentrations of zinc chloride and nickel chloride at 32 gL⁻¹ and 4 gL⁻¹, respectively. The Hull cell patterns exhibiting the effect of boric acid are shown in Fig. 1(f). Lower concentration of boric acid (<10 gL⁻¹) gave bright deposit in the current density range of 0.5-3 Adm⁻². At a concentration of 20 gL⁻¹, boric acid gave mirror bright deposit in the current density range of 0.5-4 Adm⁻². Hence, the concentration of boric acid was fixed at 20 gL⁻¹ as optimum in the bath solution. The relation between boric acid and Ni content is illustrated in Fig. 2 (e).

Effect of pH and temperature

The pH of Zn-Ni alloy plating bath had great effect on bright current density region on Hull cell cathode. The results of Hull cell experiments showed that, when the plating solution of the optimum composition was at pH of 3, bright plating layer was obtained. The pH value was buffered mainly by additive and its ammonium salt, cathode hydrogen evolution would take place as pH was low, this led to current efficiency, covered ability and brightness degree decreased. The plating layer became dark at very high pH, as seen from Fig. 1(g) and also the relation between pH and Ni content is illustrated in Fig. 2 (f).

Surface analysis of plating layer showed that species coordination agent system was most suitable to be used

at temperature range from 293-303 K. Higher temperature could increase the electric current density, and more serious cathode hydrogen evolution, it made evaporation of plating solution more faster than before, as a result, cathode current efficiency and deposition speed decreased respectively, these made internal stress of plating layer increased. Hull cell patterns showing the effect of temperature are shown in the Fig. 1(h) and also the relation between temperature and Ni content was illustrated in Fig. 2(g).

Effect of current density

There was relationship between deposition speed and cathode current density. The Hull cell experiments were carried out at different cell currents (1-4A) for 5 min using optimum bath solution. The Hull cell patterns are shown in Fig. 1(i). It was found that at a cell current of 1A, the deposit was bright in the current density range of 0.5-4.5 Adm⁻². At a cell current of 2A, the deposit was bright in the current density range of 1-5 Adm⁻². At cell currents of 3A and 4A the Hull cell panels showed burnt deposit on the side of high cathode current density region, because high cathode current density made anode polarization action increase, this led to largely decrease of deposition speed of Zn-Ni²². This observation revealed that the bath gave bright deposit in the current density range of 0.5-5 Adm⁻² and also the relation between current density and Ni content is illustrated in Fig. 2(h).

Current efficiency and throwing power

Current efficiency and throwing power were measured at different current densities using optimized bath solution. At a current density of 1 Adm⁻², the current efficiency was found to be 88%. At a current density of 2 Adm⁻², the current efficiency was found to be 92%. With increase in current density above 3 Adm⁻², the current efficiency was found to be decreased and at 5 Adm⁻² it was 89%.

Throwing power of the bath solution was measured using Haring and Blum cell at different current densities using optimized bath solution. The variation of throwing power with current density is given in Table 2. Throwing power varied from 21-24%.

Polarization studies

The potential of the cathode was measured under galvanostatic condition, at different current densities with respect to saturated calomel electrode using the bath solution with and without addition agents.

Table 2—Current efficiency and throwing power for optimum bath solution at different current densities

Current density (Adm ⁻²)	Current efficiency (%)	Throwing power (%)
1	88	23
2	93	24
3	93	23
4	92	22
5	89	21

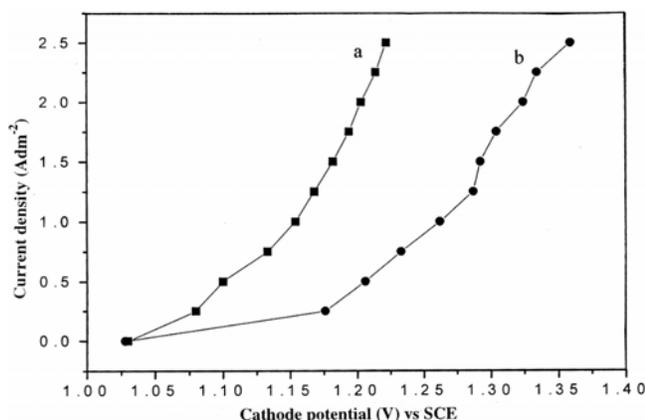


Fig. 3—Effects of addition agents on cathodic potential. {a, ZnCl₂ (32 gL⁻¹) + NiCl₂ (4 gL⁻¹) + H₃BO₃ (20 gL⁻¹) + NH₄Cl (220 gL⁻¹) [BB]; b, BB + THC–FFL (20 mL⁻¹)}.

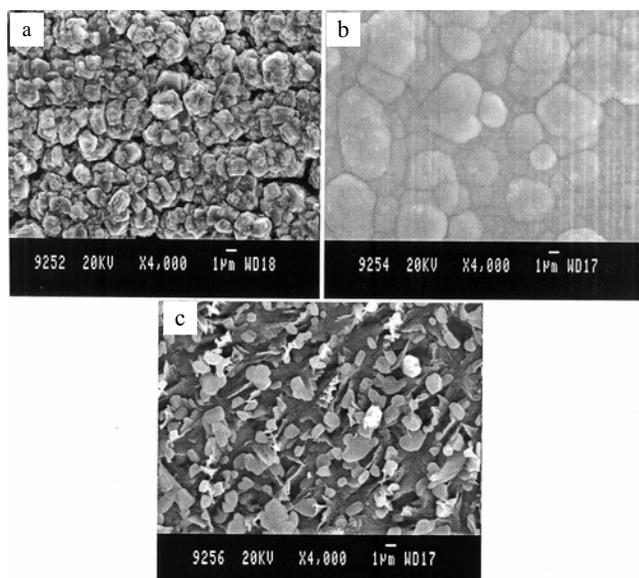


Fig. 4—SEM photomicrographs of the deposits obtained at 2 Adm⁻² in the presence and absence of addition agents at 298 K: (a) basic bath (BB), (b) optimized bath and (c) passivated deposit.

Cathodic polarization curves show that condensation product introduced into a chloride solution change the

electrode polarization (Fig. 3). In our view, varying the voltage within the studied range should not significantly affect the adsorption of condensation product and consequently change the electrode polarization. On the other hand electroactive functional groups of the condensation product adsorbed on the electrode surface can reduce the surface stress at the gas-electrolyte interface. Hence, the decrease in the hydrogen evolution over potential at low voltage can be attributed to an easier detachment of hydrogen bubbles from zinc electrode in presence of additives, which leads to an increase in the active electrode surface. In this case, the electrolysis conditions approximate steady-state conditions that promote the bubble growth to critical size rather than their nucleation. With increasing voltage, the ratio between the rates of bubble nucleation and its growth will increase. In other words, the number of adsorbed hydrogen bubbles will be greater, but their size will be smaller. Therefore, the number of hydrogen bubbles of critical size capable of detaching themselves from the electrode will be smaller.

At high voltage, the surface-active effect of additives is weaker, and hydrogen evolves at the cathode whose surface area does not change due to the bubble detachment. On the other hand, such a gas film comprising a large number of small bubbles increases the electrode solution interface resistance and, consequently, the cathodic polarization. This conception agrees with the data obtained at a high condensation product concentration^{24,28}.

Surface morphology and FTIR studies

The nature of crystal growth in the presence and absence of addition agent is explained with the help of SEM photomicrographs and is shown in Fig. 4. Figure 4(a) is the SEM photomicrograph of the deposit obtained from the basic bath shows coarse-grained deposit having irregular crystal size. SEM photomicrograph of deposit obtained from the optimum bath shows perfect crystal growth, uniform arrangement of crystals, refinement in crystal size and hence bright deposit is as shown in Fig. 4(b). Fig. 4(c) is the SEM photomicrograph of passivated deposit.

The FTIR spectrum (Fig. 5) which was obtained from optimum bath solution (Table 3) shows an absorption peak at 1588 cm⁻¹ which may be assigned to C=N stretching and peak at 1503 cm⁻¹ corresponds to N-H bending and band at 1384 cm⁻¹ and 1335 cm⁻¹ correspond to C-N stretching frequencies. The

absorption peaks in the FTIR spectrum revealed inclusion of addition agent in the deposit during electrodeposition.

Corrosion resistance

The Zn alloy deposited panels were taken out after every 24 h of test, dried and inspected visually. Table 4 summarizes the results of corrosion studies by salt spray in 5% NaCl solutions. The plates deposited from basic bath and optimum bath solutions without

Table 3—Optimum bath composition and operating conditions

Bath composition (gL ⁻¹)	Quantity	Operating conditions
ZnCl ₂	32	Anode: Zinc metal (99.99%)
NiCl ₂	4	Cathode: Mild steel
NH ₄ Cl	220	Temperature: 293-303 K
H ₃ BO ₃	20	Bright current density range: 0.5-5 Adm ⁻²
Condensation product (THC-FFL) (mLL ⁻¹)	20	Agitation: Air

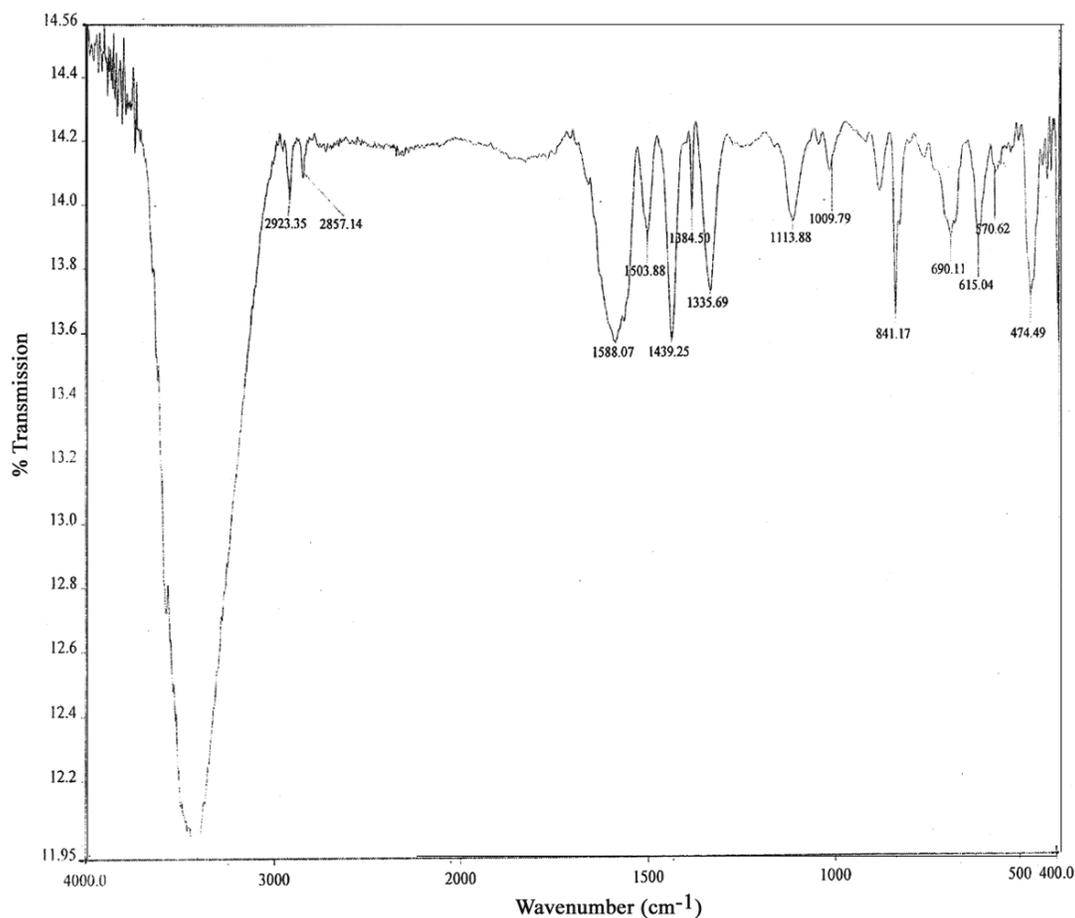


Fig. 5—FTIR spectrum of the scrapped zinc deposit obtained from optimum bath.

Table 4—Visual observation of white rust on Zn-Ni deposits during salt spray test

Bath system/ Hours	Zn-Ni deposit from basic bath		Zn-Ni deposit from optimum bath	
	Un passivated	Passivated	Un passivated	Passivated
24	NR	NR	NR	NR
48	R	NR	NR	NR
72	-	NR	NR	NR
96	-	R	NR	NR
120	-	-	NR	NR
144	-	-	R	NR

R = Rust formation
NR = No rust formation

passivation showed white rust within 48 h and 96 h of testing respectively but the passivated deposit from optimum bath solution did not show white rust even after 144 h of testing. This test indicated the pore-free nature of the deposits obtained in presence of addition agents.

Metallurgical properties

Standard bend test was used to measure both adherence and ductility of Zn-Ni deposits. No crack or peel off in the deposit was noticed even after 180° bending. This indicated good adherence and ductility of Zn-Ni deposit on steel. The microhardness of Zn-Ni alloy was found to be 130 VHN.

Consumption of brightener

The amount of condensation product consumed for 1000 amp-h was 5 mL⁻¹.

Scaling up study

The commercial applicability of the developed bath was explored by performing the plating experiments in a vat of 25 L capacity. The bath solution with optimum concentration of bath constituents was prepared. Steel components of different sizes and shapes (plates, rods, nuts, bolts, small pipes, clamps etc) were degreased, electrocleaned and given acid dip followed by water wash. These treated components were rigged by copper wire and connected to the negative terminal of the DC power supply. Electroplating was carried out at different current densities (2-6 Adm⁻²) with and without agitation. The components after deposition were removed from the plating vat and subjected to bright dip and passivation. The passivated articles were subjected to corrosion resistance test in salt spray chamber. The components of irregular shapes plated under stirred and unstirred conditions showed no white rust. This indicated the ability of the bath to produce uniform deposit on the component of the irregular shape.

Conclusion

By comparing the results of this study and those of other literature, synthesized compound was found to have analogous properties to cyanide, very well stability to heat and pH, and fine surface activity properties, as high stability of its coordinated ion, moreover, the plating solution is very stable. The alloy plating layer which contains Ni 9-10 wt%, is acquired by electroplating of Zn-Ni alloy with the additive. The results show, that exterior of plating layer is smooth and bright, besides this, the plating

solution has high current efficiency and throwing power and also the plating solution has simple component and nice ability of deep plating. Also the developed compound is non-toxic, so it is very promising to be put in to large-scale production.

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