Available online at www.journalijmrr.com

INTERNATIONAL JOURNAL OF MODERN RESEARCH AND REVIEWS

ISSN: 2347-8314

Int. J. Modn. Res. Revs.
Volume 3, Issue 1, pp 575-578, January, 2015

ORIGINAL ARTICLE

ELECTROCHEMICAL DEPOSITION OF NICKEL FROM ELECTROLYTES CONTAINING ADIPIC ACIDBUFFER

[1]S.Manikandan, [1]M.Maria Berjilia and [2]K.B.Dhanalakshimi

[1]Government Arts College-Ariyalur, Tamilnadu India-621 704.
[2]Government Arts College-Musiri, Tamilnadu India-621 211.

Article History: *Received 12th December,2014, Accepted January 27th 2015, Published 28th January,2015*

ABSTRACT

Electrodeposited nickel, prepared from a sulphate bath containing adipic acids in place of a boric acid buffer was studied under different current densities, PH and temperature. To judge the quality and nature of deposit at various concentration of adipic acid, a Hull cell was employed and operated at Lampers cell current for 5 minutes. The current efficiency and throwing power of the adipic acid buffered nickel electrolyte were investigated under various conditions and compared with the conventional watts nickel electrolyte. The optimum conditions of deposition were established and it was shown that replacing the boric acid buffer by the adipic acid result in better properties of the batch and the deposits. It was found that the throwing power of the adipic acid buffered electrolyte was improved than watts nickel bath where as the current efficiency was marginally influenced. The corrosion behavior of the nickel deposit was studied in 3.5% Nall solution using Potentio dynamic Polarization and electrochemical impedance spectroscopy. The corrosion data revealed that the adipic acid buffered nickel deposit is more corrosion resistant than watts nickel deposit. The surface morphology was studied with SEM and XRD.

Keywords: Nickel; Coating; Electrodeposition; Adipic acid

1.INDRODUCTION

Electroplated nickel has found widespread applications as decorative as well as functionally suitable metal coatings. Numerous studies have shown that the physico mechanical properties of the nickel coatings depend strongly on the deposition parameters and the bath composition [1,2]. In this work the watts bath was used, i.e. containing boric acid as the main buffer component. Recently, new buffering cononents, mainly organic acids, have been extensively studied. Because they offer higher buffer capacity than the boric acid does [3,4]

Among all the studies, only a few have addressed the corrosion properties of electrodeposited nickel prepared from a watts bath [4,5]. Electrodeposited nickel shows an improvement in corrosion resistance with decreasing grain size [6], on the basis of the analysis of the microstructure of electrodeposited nickel, the corrosion behavior was analyzed in 3.5% Nall by potentio dynamic polarization.

The main aim of the present work was to examin the new electrolytes for nickel plating, which contains Adipic acid (for comparison) instead of the boric acid (BA) as the buffer. The Adipic acid was chosen because it is highly soluble in the nickel plating bath.

**Corresponding* author: *S.Manikandan, Government Arts College-Ariyalur, Tamilnadu India-621 704*

2. EXPERIMENTAL :

Nickel was deposited as 60° C (or 40° C) from the plating bath containing reagent grade constituents. The batch compositions are shown in Table 1. The nickel salts and boric acid were supplied by path (Poland), the other components were supplied by Aldrich. The PH was adjusted to 5.0 or 3.0 and the temperature was maintained at 50 ° C. The applied current densities ranged from 2 to 8 A dm2). As the cathode a Nickel plate (A=1 cm2) or tip of a Nickel rod (Ar= 0.08cm2) set in a Teflon holder were used. The anode were plates of technical nickel with the geometrical area of 2 cm2. Prior to use the working electrodes. Were mechanically Polished with emery papers (down to grid 1000). All potentials were measured against saturated calomel electrode and real calculated against the standard hydrogen electrode (SHE). The deposition was carried out by the steady – state galvano static and pontentiostatic techniques. (The cathodic current efficiencies through measurements of the mass of the deposit and integral quantity of electricity as the Wt exp / Wt theo, where Wt exp is the weight of the deposit obtained experimentally and Wt theo is the theoretical weight of the Ni deposit according to Faraday's law current efficiency was measured in all studied solutions at current densities from 2 to 8 Adm2, with stirring, at 50 ° C.

The throwing power of the watts Nickel Adipic acid buffer plating bath was determined using a Haring Blum cell [7]. This is a long and narrow cell in which two cathodes are placed on two sides of the anode and mild steel cathodes of size of the anode and mild steel cathodes of size 5cm x 2.5 cm. The panels were mechanically polished, degreased using trichloro ethylence, cleaned using an alkaline solution, washed and pickeld with 10% H2S04, washed in tap water, rinsed with distilled water and dried, the unwanted side of the panels was covered with cellphane tape and one of the cathodes was nearer to the anode than the other. The distance ratio was 1:5. The panels were take out washed and rinsed with water dried and weight.

The percentage of throwing power can be calculated from Eq(1)

Percentage of throwing power = $\frac{L - M}{L+M-2} * 100$ Where 'L' is the Lionear distance ratio and 'M' is the

metal ratio.

These are defined as Liner ratio (L) = $\frac{\text{Distance of a far cathode}}{\text{Distance of near cathode}}$

Metal ratio (M) = $\frac{\text{Weight of deposition near catchode}}{\text{Weight of deposition on far cathode}}$

The hardness values of the watts nickel Adipic acid buffer deposits were measured using a microhardness tester in Vickers pyramid numbers. The electrochemical impedance spectros copy (EIS) experiments were carried out at the dc deposition Potential with superimposed ac voltage of amplitude. 10mv in the frequency range of 100 mhz to 100 khz using EG & G impedance analysor, medel 6310 software model M270. The coated samples and Platinum foils were used as the working and counter electrodes respectively. The SCE was used as the reference electrode. The 5 % Nal solution was used as the electrolyte medium.

The real part (z) and the imaginary part (z") of the cell impedance were measured for various frequencies. Impedance measurements were carried out bath watts nickel adipic acid buffer and different concentration in deposits.

Microhardness measurements were carried out using a Vickers microhardness tester, applying 50 g to 100 g load for 5s time [8]

The morphology of the deposits was examined by scanning electron microscopy (SEM)

3. RESULT AND DISCUSSION

Effect of additive in Hull cell measuremtns:

A 267ml Hull cell was made of PVC was used for studying the effect of additives on the deposit characteristics at various current densities [a]. The concentration of the additives was varied from log/ l to 30g / l in the standard Adipic acid buffered bath. The optimized concentration and nature of the deposits are presented in Table 2 and in Fiog.1.

By comparing this deposit patterns, it can be concluded that 20g/l adipic acid gave a wide range of semibright deposits. Hence this bath was used for further deposition studies.

Table 1: Adipic acid buffered concentration data

Conc	Current efficiency
10	78.03
20	80.90
30	81.78
40	69.18

Table:2Current density ranges for various deposits obtained with different additives in Adipic acid buffered bath.

Additives	Concern (g/l)	Nature of deposits	Current density ranges(A/ dm2)
Nickel sulphate	250	dull	2.8
Nickel chloride	45	Semi-bright burnt	6.0 Above
Boric Acid buffer	35	dull	8
Adipic acid	10	Semi-bright	5
Adipic acid	20	Semi-bright	0.8
Adipic acid	30	Semi-bright	2.8

The effect of operating parameters such as temperature 50° C and PH 4. The deposit pattern for this bath was obtained by passing a cell current of 2 amperes for 5 minutes.

Effect of adipic acid buffered and watts Nickel in haring – Blum cell measurements

Throwing power of the watts nickel solution containing 250g/l Nickel sulphate, 45g/l Nickel chloride, 35g/l boric acid was determined using a haring blum cell. There was made PH 4 and at 50° C , for current density from 2 to 8 A/dm2. In place of boric acid buffer, 20g/l adipic acid was added and the throwing power of the solution was studied under same experimental conditions described as above. Thus presence of adipic acid improves the throwing power of the nickel bath in Table 3.

Table 3:Throwing power data in watts nickel and adipic acid buffered

A/dm2	Watts nickel	Adipic acid buffer
2	13.0534	17.8361
4	11.0833	11.8361
6	11.6585	12.4707
8	6.6737	8.5152

Effect of watts nickel and adipic acid buffer in current density and current efficiency:

The current efficiency of the new electrolyte containing nickel sulphate 250g/l, Nickel chloride 45g/l and adipic acid was determined at various current density, PH and temperature. The amount of adipic acid added to the watts bath was varied at 10 to 40g/l. For comparative study the watts nickel bath. The variation of current efficiency with amount of adipic acid was determined at 10 to 40g/l adipic acid the PH4 and temperature 50° C and 4A/dm2. The above 30g/l adipic acid concentration 69.2% current efficiency decreased considerably attained further studies 20g/l adipic acid concentration, which had 81.8%. The variation of

current density of the nickel deposition was varied 2 to 8A/dm2 the PH4 and temp-50° C up to 6A/dm2 the current efficiency increased significantly and reached a maximum of 82.6% in the above current density, the current efficiency decreased to 78.6% at 8A/dm2 (Table 4)

Table.4: Effect of watts nickel and adipic acid concentration in current efficiency and current density

Current density (A/dm2) watts Ni	Current Efficiency	Current density(A/dm2)Adipic Acid	Current Efficiency
2	70.81	2	81.11
4	80.89	4	96.27
6	82.60	6	94.23
8	785.68	8	92.80

The current efficiency of the new nickel electrolytes containing adipic acid buffer is compared with the conventional watts nickel electrolyte. The effect of current density on current efficiency of the watts nickel electrolyte at temperature 50° C and PH4. The 4A/dm2 the current efficiency was maximum 99.2%. The above this current density the current efficiency is maintained at 94+ - 2%.

Microhardness studies of watts nickel and adipic acid buffer deposit

The hardness results in the presence of the watts nickel and adipic acid concentration are presented in Table 5. The Plating bath control the quality at the deposit which is related to the hardness. Hence the microhardness studies assume importance in the present investigation.

Table 5:Microhardness data of watts nickel and adipic acid buffer

Watts nickel and Adipic acid buffer (A/dm2)	Hardness HN50 Watts Ni	Hardness HN50 Adipic acid buffer
2	175.27	133.03
4	246.93	168.17
6	187.80	141.40
8	166.10	123.09

The microhardness of the nickel deposit obtained at various current density was measured at 50g load using an indentation technique. The harndess of the nickel deposits obtained using adipic acid buffered nickel bath is comparatively higher by 1 ½ times at all current density ranges. It can also be seen that the hardness value increased current density up to 4A/dm2 and then decreased with further current density. The adipic acid buffered bath had more hardness of above 1 ½ time more than the watts nickel bath.

Potentiodynamic Polarization and impedance measurements:

Corrosion behaviour of the nickel deposit prepared from adipic acid buffered bath was determined in 3.5% Nacl solution using potentiodynamic polarization method. The thickness of the deposit was 10mm. The tangents were drawn on the polariztion curves. At the point of intersection, Ecorr and Icorr values are obtained. Also the anodic and cathodic slopes are measured. The data are given in table 6. The results were compared with steels substrate and nickel deposit prepared from watts nickel and adipic acid buffered bath. The deposit from the watts nickel and adipic acid buffered bath. The deposit from the watts nickel and adipic acid buffered nickel had lowered corrosion currents (3.5 and 4MA/cm2) lowerd corrosion currents than the steel substrates (22.3) hence it can be concluded the nickel deposits protect the steel substrate against corrosion in chloride environments.

Impedance measurements:

The impedance results of steel and the nickel deposits obtained from watts nickel and adipic acid buffered nickel are presented in table 6. The Nyquist plots clearly gave semicircles from the plots, charge transfer resistance value (Rct) were calculated. It is known that Icorr is inversely propostional to the charge transfer resistance

Icorr 2 1/Rct

Comparison to steel, the nickel deposits had more Rct vakyes indicating a better corrosion resistance offered by the nickel deposits. Hence the nickel deposits obtained from the adipic acid buffered bath protect steel more watts nickel.

Table 6:Corrosion data using potentiodynamic polarization and impedance data.

Sample	Ecorr(mv Vs scE)	Icorr(MA/cm2	Rct(ohms)
Steel	-712	22.3	700
Watts nickel	-415	3.5	900
Adipic acid buffered nickel	-504	4	1300

Morphology:

The SEM images of the nickel deposit at various magnification. The structure is watts nickel deposited at 1000 and 3000 magnification. The size of the crystal is found to be about 1 micrometer. At higher magnification a pyramidal growth is found. The adipic acid buffered nickel deposited at 1000 and 5000 magnification. The adipic acid buffered structure is not modified, with increasing the current density in figure? The EDAX speetrum analysis reveals the presence of nickel peaks in the deposits shown in figure?

The three dimensional structure was observed at the adipic acid buffered nickel deposit, using an atomic force microscope (AFM). At various scanning area is adipic acid buffered deposit. The scanning area increase the roughness of the deposit is seen in to increase.

The orientation of the watts nickel deposit and adipic acid buffered nickel deposit is obtained with adipic acid buffered nickel deposit is obtained with X-ray diffractometer. The peaks corresponding various position is shown in figure and the XRD pattern in table 7. The XRD patterns clearly indicate that (iii) plane is the prepared orientation in both cases the most preferred orientation (iii) in both cases.

Table 7 XRD data of Nickel deposit

				Adipic acid buffered Ni deposit	
Watts Nickel deposit					
Pos.(2 Theta)	**d-pacing(Ao)**	**hk1**	**Po(2 Theta**	**d-spacing(Ao)**	**hk1**
44.9647	2.01439	111	44.9820	2.01365	111
52.3633	1.74584	200	52.3690	1.74567	200
76.8296	1.23972	220	76.8299	1.23971	220

4. CONCLUSION

As of now, boric acid is being used as the buffer in Nickel electroytes. As a substitute for boric acid, many organic acids had been tried now and then. In this study a systematic attempt is done using adipic acid as the buffer. The results of the study are as follows.

From the Hull cell study results, it was concluded that 20g/l of adipic acid gave a semibright deposit between 2 and 6A/dm2 at 50° C and PH4. However watts nickel bath gave about 94 to 96% current efficiencies at the same operating conditions. The throwing power of the adipic acid buffered nickel was found be higher than the watts nickel bath. Adhesion of the nickel deposit to the steel substrate was found to be very good. the adipic acid buffered nickel deposit had about 11/2 times more hardness than the watts nickel deposit potentiodynamic polarization and impedance studies for characterizing the corrosion resistance of the nickel deposits clearly revealed that the corrosion resistance was almost the same as that of watts nickel deposit. Thus both nickel deposits are equally better in protecting the steel substrate. The structure of the nickel deposits revealed the uniform and fine-grained deposit with (iii) as the most preferred orientation.

5.REFERENCES:

Abd, E.A. EI Meguid, S.S. Abd EI Rehim, E.M Moustafa, .1999.Trans. Inst. Metal Finish 77,188.

Shahin, G.E.2001. Proceedings ALSF Nickel Chromium Focus, February 1,Orlando, P-48.

Yang, C.2001.Plating and surface finishing, 88(5):116.

Saitou, M.2007.Journal of the Electrochemical society, 148(12).

Ibrahim, M.A.M., S.S Abd EI Rehim, S.M.Abd. EI Wahaab, M.M Dankeria.1999.Plat. Surf.Fin.86 ,69.

Gamburg, Yu.D;M.yu Grasheva, S.Biallozor, M.Hass,2002. Surf. & coat. Tech. 150, 95-100

Nohse 1966.(watter) (Ed) Investigation of Electroplating and related solutions with the aid of hullcell, Robert Draper Ltd, Teddington .

Tan, A.C. 1993.(Ed), Tin and solder plating in the semiconductor Industry, chapman & Hall, London.

.Mayanna, S.M ., B.N. Maruthi. 1996. Metal Finish 94,42.

*****