PARAMETRIC OPTIMIZATION OF ELECTROLESS NI-P COATING USING L_9 ORTHOGONAL ARRAYS

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Abstract - Coatings are one of the rising techniques to stop degradation of metals' surface from corrosive pitting and wear. Electroless Ni-P coatings were developed and successfully deposited over Copper substrate.The coatingwas varied by varying the concentration of the metal source, Nickel Sulphate and the reducing agent, Sodium Hypophosphite.The corrosion rates of all the coatings were measured using Tafel Tests. Taguchi modelling was done with the help of L_9 orthogonal arrays with corrosion rate as the response parameter. The optimized sample was obtained based on the Signal to Noise ratio of individual levels of the coatings. The level 1 and level 3 of the above mentioned factors were the optimized parameters. The optimized sample was characterized using Energy-Dispersive X-Ray Analysis, Scanning Electron Microscopy. Energy-Dispersive X-Ray Analysis showed the Ni-P coating was a medium Ni-P coating.

Index Terms - Coating, Taguchi, Signal to Noise Ratio, Scanning Electron Microscopy.

I. INTRODUCTION

Corrosion is one of the primary phenomenon which is responsible for the degradation of material and its surface. Corrosion causes the metals surfaces in machineries to form a more stable compound by reacting with the surface to which it was exposed to [1-3]. This causes the decrease in the predicted life of various machines. Hence replacement of parts will be mandatory, increasing the net cost of the product being manufactured. In order to improve the running life of various parts of the machines in various industries several techniques include various coating techniques.

Electro less coatings first came into being in 1946. This technique coats the substrate sample with the help of a bath. This bath is called an electroless bath. The bath comprises of a source of metal which has to be deposited, a reducing agent which reduces the metal ion to its ground state, complexing agent which forms a complex with the metal ion and controls the deposition rate and a buffering agent which maintains the pH of the solution [4-6]. Since reduction process involves the production of H^+ ion in the solution, the pH of the solution tends to change[7-8].Electroless coatings is an economic coating as there is no external source of energy required. Due to the use of complexing agentthe coatings are very uniform, allowing any non-uniform surface of the substrate to form a plane surface by coating.

In this present study we have developed a protective layer of Ni-P over Copper substrate.Ni-P coating has good anti-corrosion properties [9]. The corrosion properties gets affected by the weight percentage of phosphorus. The anti-corrosion characteristics were studied using electrochemical tests. The corrosion rate was measured for the coating. The coating characteristics are ought to vary if we vary the concentration of metal source, Nickel Sulphate and reducing agent, Sodium Hypophosphite as they determine the weight percentage of Nickel and Phosphorus in the coating. Hence we varied the concentrations in three different levels and tried to optimize the coating with optimum corrosion characteristics. A L_9 orthogonal array design was used in Taguchi modelling to optimize the parameters based on the corrosion response of the samples.
The optimized coating was characterized using Energy Dispersive X-Ray Analysis. The surface study of the samples was done Scanning Electron Microscopy.

II. EXPERIMENTAL DETAILS

2.1 Substrate Preparation

Rectangle-shaped (20×15 mm^2) copper plates with 0.1 mm thickness were used as the substrate material for deposition of electroless Ni–P coatings. The surface of the copper substrate was pre-treated in order to remove any hydrocarbons, oxide layers, surface asperities and dust particles from the surface. In order to accomplish this the substrate were first cleaned with acetone, followed by rinsing with distilled water for 1-2 minutes. The surface of the substrate was cleaned with 1N HCl for 2 minutes. It was again rinsed with distilled water for 2 minutes. Activation is also a very important step of sample preparation. Samples were activated with Palladium chloride solution. It was dipped for 8-10 seconds at 55 °C followed by rinsing with distilled water for few seconds. The surface activated samples were immersed in the electroless bath.

2.2. Coating Preparation

The coating parameters are mentioned in table 1. The Nickel source is Nickel Sulphate. Tri-sodium Citrate Dihydrate was used as the complexing agent. The complexing agent forms a stable complex, Nickel Citrate Hydrate, which releases the metal onto the surface of the substrate. Ammonium Sulphate acts as the buffering agent. The pH of the solution was maintained at 5. The reducing agent used was Sodium Hypophosphite. The temperature of the setup was maintained at 85□C. The deposition took place for 90 minutes.

Table 1. Chemical composition and the agents of the Ni–P electroless bath

Factors	Parametric Value
Nickel Sulphate Hexahydrate	20 /25/ 30g/L
Sodium Hypophosphite	16/20/24 g/L
Trisodium Citrate Dihydrate	15 g/L
Ammonium Sulphate	10 g/L
Temperature	80/ 85/ 90^{o}C
pH of solution	5
Deposition Time	90 minutes
Activation temperature	55^{o}C
Bath Volume	250 cm^3

2.3. Corrosion Tests

The electrochemical corrosion analysis of the samples is evaluated by electrochemical impedance spectroscopy (EIS). Electrochemical tests were done in a 3.5 wt.% NaCl aerated aqueous solution using a classic three electrode cell with a Pt-wire as counter-electrode and an Ag/AgCl electrode (q207 mV vs. SHE) as reference. The sample is covered by polyester resin and certain area is left uncovered approximately 1.5 cm for testing series and parallel resistance of the sample.

2.4.Design of Experiment

Design of tool is an impressive tool to determine the optimized samples. The modelling was done using Taguchi method. Taguchi method uses orthogonal arrays for the execution of optimization. Orthogonal arrays gives the minimum number of experiments that needs to be carried on in order to optimize the sample. L_9orthogonal array model uses two factors at three different levels. All the possiblecoating combinations with the variation of the factors in three different levels were prepared.

The modelling is based on the concept of Signal to Noise ratio. Optimization of the coatings' parameter using S/N ratio can be done in three ways. We pursue the smaller the better technique as the response of the coating corrosion rate is required to be as minimum as possible. The S/N ratio is the ratio of the wanted response to the unwanted signal in the design. The S/N ratio is calculated using the following formula:

$$S/N= -10\ \log_{10}(\frac{1}{n} * \sum y^2)$$

Where n is the experiment number and y are the responses till that experiment.

2.5. Surface Study and Characterization of the Optimized sample

Surface Morphology of the surface of the coating was determined by the Scanning Electron Microscope (SEM) analysis. The SEM analysis was done on the SOF software in JEOL-Jsm 7610F machine. X-Ray Diffraction (XRD) analysis was done on the same software on the same machine to determine the phase present in the coating. In order to find the weight percentage of different elements present in the coating Energy Dispersive X-ray Analysis (EDAX) was done. It was done on AZTEC software using OXFORD X-max 50 machine.

III. RESULTS AND DISCUSSIONS

3.1 Corrosion Rate

The corrosion rate was determined in the Tafel tests. The atomic mass was taken as 55.845 g and density as 7.874. The valence was taken as 2. Putting these as the initial parameters the corrosion rate was calculated. The corrosion rate for each coating is shown in table 2.

Table 2: The corrosion rate of each coating.

Experiment Number	Nickel Sulphate (g/L)	Sodium Hypophosphite (g/L)	Corrosion Rate (µm/Year)
1	20	16	1.94
2	20	20	1.21
3	20	24	0.98
4	25	16	2.03
5	25	20	1.49
6	25	24	1.32
7	30	16	2.17
8	30	20	1.56
9	30	24	1.19

3.2 Taguchi Analysis

Since S/N ratio is the ratio of the expected response to the unwanted response, higher value of S/N raio will suggest better response. The S/N ratio for each level for the two factors are shown in table 3. It can be observed that level 1 of Nickel Sulphate and level 3 of factor Sodium Hypophosphite had the maximum S/N ratio values and were the optimized paramters. The nature of the S/N ratio for Nickel Sulphate is decreasing in nature with increasing values of Nickel Sulphate. An opposite nature is observed in the case of Sodium Hypophosphite as shown nin figure 1.

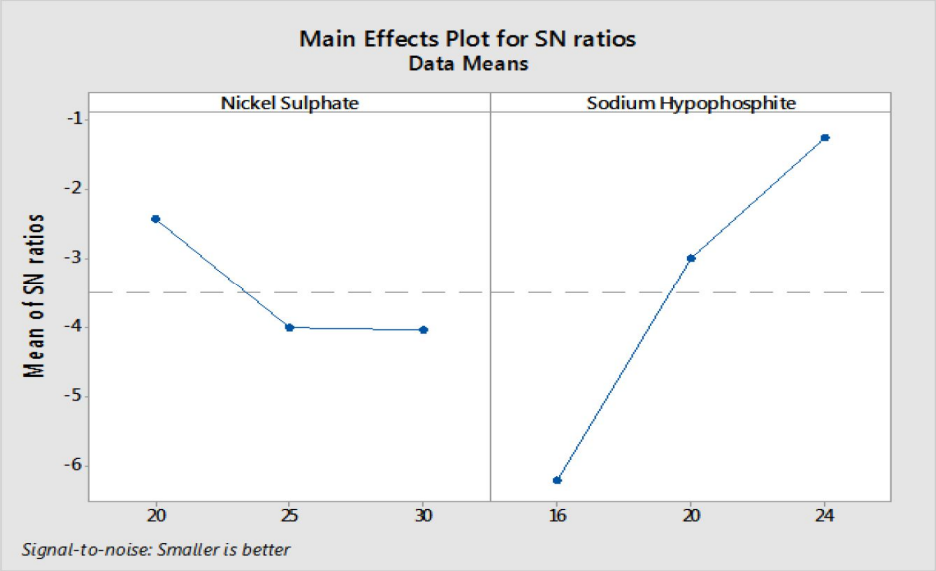


Figure 1: The S/N ratio plot of different levels of the two factors

Table 3: S/N ratio at each level for different factors

Level	Factor 1	Factor 2
	Nickel Sulphate	Sodium Hypophosphite
1	-2.412	-6.212
2	-4.008	-2.994
3	-4.034	-1.249
Delta	1.622	4.963
Rank	2	1

Hence the optimized parameters from figure 1 and table 3 are 20 g/L of Nickel Sulphate and 24 g/L of Sodium Hypophosphite.

3.3 Surface Study and Characterization

Figure 2 shows a compact coating has taken place as no holes are shown on the surface. The porosity of the coating will be low as well. The image shows uniform distribution of suspended agglomerated particles on the surface, proving that the copper substrate exhibited uniform coating.

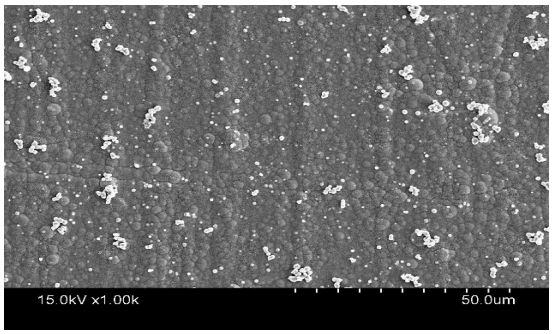


Figure 2. Scanning Electron Microscope Image of the optimized Ni-P Coating

The percentage of Nickel and Phosphorus deposited on the surface was determined using EDAX. The weight percentage of Nickel was 94.55 % and Phosphorus was 5.45%.The weight percentage of 5.45% of phosphorus determines that the coating is a medium Phosphorus coating.

CONCLUSION

The following results can be concluded:

- Electroless Ni-P coating was successfully deposited
- Taguchi Mathematical Modelling suggested the coating with 20 g/L of Nickel Sulphate and 24 G/L of Sodium Hypophosphite were the optimized paramters.
- The optimized coating had the minimum corrosion rate of 0.98 mm/Year.
- EDAX showed the optimized coating was a medium Ni-P coating

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