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Review Article

Optimization of Bath Parameters for Electroless Nickel Plating Using Fractional Factorial Design

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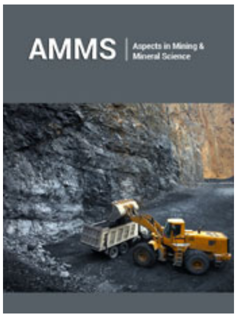

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Abstract

Electroless nickel coating is a revolutionary coating process that can be effectively used to cover a spectrum of alloys and composites, each having its own features. Electroless nickel coatings have been primarily utilized for purposes of both corrosion resistance and wear resistance. Additional qualities, such as overall smoothness of the deposit, low friction, acceptable plating rate, and both electrical properties and magnetic properties, makes them an appropriate choice for a wide variety of applications. Characteristics of the electroless nickel coating are essentially determined by both constituents of the electroless solution, and the conditions used during deposition. Bath temperature, nickel source concentration, and pH of the solution are three key deposition parameters. Furthermore, heat treatment does tend to alter microstructure of the coating and thereby exert an influence on hardness, wear resistance and corrosion resistance. In recent years, researchers have published their findings pertinent to an evaluation of electroless nickel coating on the performance of various types of substrates based on hardness, roughness, corrosion resistance, friction, and wear resistance. Several viable ways aimed at solving the challenges specific to parameter optimization have been put forth in the published literature. In this research study, the electroless coating process was performed based on fractional factorial design. The optimal coating parameters were determined using the Analysis of Variance (ANOVA) approach. The findings reveal bath composition can be used to optimize thickness of the coating.

Keywords: Electroless nickel plating; Design of experiments; Analysis of variance; Interaction plots

Introduction

The method of making a nickel alloy by pouring a water-like solution onto the surface and without the use of an electric current is referred to as electroless plating of nickel. Electroplating, on the other hand, relies on direct current as the external source to ensure that the nickel ions in an electrolyte react with the nickel metal at the level of the substrate to form a plating on the nickel metal. Electroless nickel plating is a chemical method that essentially uses chemical reduction to convert the nickel ions in solution to the metal nickel. Sodium hypophosphite is a commonly used reducing agent. Both sodium borohydride and dimethylamine borane are viable alternatives, though they are not used as often as they should be. More than 99 percent of all electroless nickel is made using sodium hypophosphite. Electroless nickel often finds for itself use in a spectrum of industries due in essence to a combination of unique qualities that include the following:

(a) Homogeneity in thickness,

(b) Surface hardness,

(c) Magnetic responsiveness, and

(d) Corrosion resistance.

Despite these notable advantages, not all designers, engineers, metallurgists (materials engineers), and others involved in the development of a product are equally qualified to use electroless nickel [1]. Nevertheless, in recent years this plating technique has gradually grown both in stature and significance to become well-established as a functional coating in the industries specific to

(i) Food grain manufacturing,

(ii) Electronics,

(iii) Electrical,

(iv) Oil and gas,

(v) Chemical,

(vi) Aerospace, and

(vii) Automotive

Others have noticed and put to effectively use the benefits offered by electroless plating for a spectrum of applications. Conventional plating, often known as 'electroplating', is a process of reducing the metal ions to their metallic state and subsequently depositing them onto the desired location. Electrical energy is often used to power the cathode. During electroless plating using an aqueous solution, a metal ion is reduced through a catalytic reaction and forms the basis for a chemical reduction process containing a reducing agent that aids in allowing the deposition to occur. Without the need of electrical energy, the metal can be shaped. A completely different stabilizer mechanism is often used in electroless nickel plating. This mechanism is made to be cost-effective [2]. This revolutionary technology offers the additional advantage of brightening metals that have a dull surface finish by producing thicknesses of the order of 100mm without the occurrence of both pitting and microscale roughness at the fine microscopic level.

Approach

The cause-and-effect diagram

Cause and effect diagram is a tool, which helps to identify the possible causes of a specific problem or a quality characteristic (Figure 1). It graphically illustrates the relationship among the factors identified, which exert an influence on the outcome(s) selected.

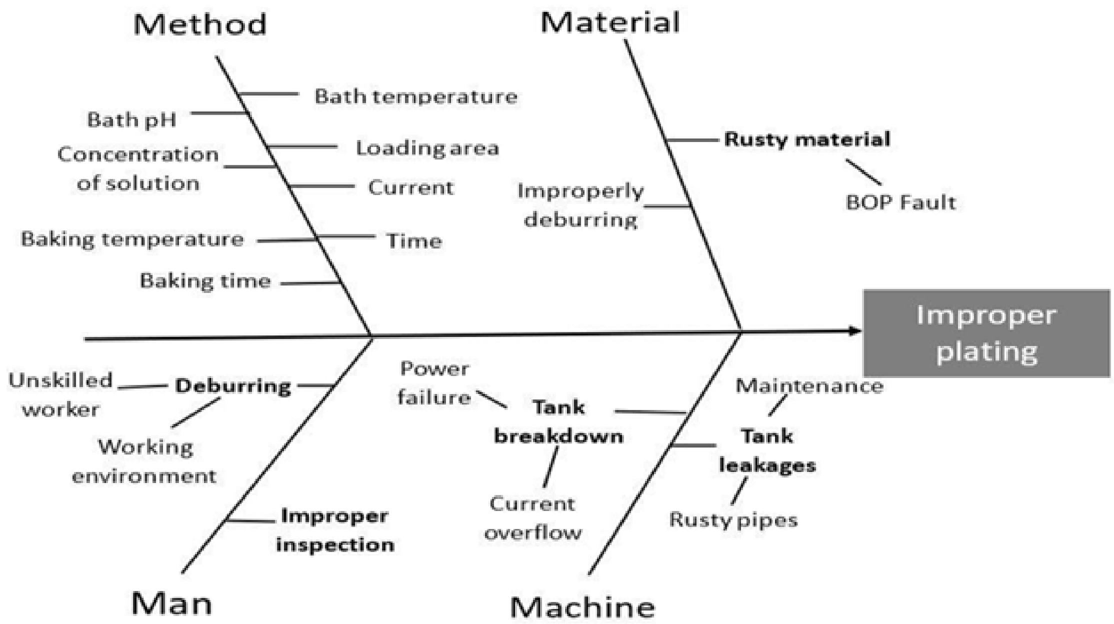

Figure 1: The cause-and-effect diagram.

Design of experiments

Based on information available in the published literature [3-7], it was decided that few experiments would be carried out by varying the following six factors:

(i) Nickel metal concentration in the solution,

(ii) Bath temperature,

(iii) Solution pH,

(iv) Plating time,

(v) Loading area, and

(vi) Heat treatment temperature.

Based on the prevailing operating situation, the number of factors was chosen to be both high and low. In this specific study, a 16 fractional factorial design with two replicates was chosen. The respective levels are provided in Table 1. There would be 16 runs based on the design chosen for two replicates. The response variable of interest was thickness of the plating.

Table 1: Factors and their respective levels.

Design Factors	Levels	
	1	2
Conc. Of solution (gm/L)	5	6
Temperature (°C)	83	93
pH	4.4	5.2
Plating time (minutes)	30	45
Loading area (mm2)	90	130
Baking temperature (°C)	300	400

Experimental procedures

The randomization order was determined using the Minitab software [8,9]. The list of randomized orders (indicated by run order) is provided in Table 2. The standard order refers to the Yates design order. The electroless nickel plating was carried out in a 1000ml electroless plating bath. The experiments were carried out in a randomized order, and the test results systematically recorded for the purpose of analysis and interpretation.

Table 2: Plating thickness of the experiment conducted.

Standard Order	Run Order	Nickel Concentration [gram/litre]	Bath Temperature [°C]	Bath pH	Plating Time (minutes)	Loading Area (cm^2)	Heat Treatment Temperature (°C)	Plating Thickness in (microns, µm)
1	5	5	83	4.4	30	90	300	4.10
2	3	6	83	4.4	30	130	300	5.63
3	14	5	93	4.4	30	130	400	8.47
4	13	6	93	4.4	30	90	400	11.60
5	16	5	83	1	30	130	400	7.75
6	12	6	83	1	30	90	400	8.80
7	2	5	93	1	30	90	300	9.85
8	4	6	93	1	30	130	300	12.10
9	11	5	83	4.4	45	90	400	7.90
10	10	6	83	4.4	45	130	400	8.65
11	1	5	93	4.4	45	130	300	13.60
12	9	6	93	4.4	45	90	300	14.90
13	7	5	83	5.2	45	130	300	11.40
14	8	6	83	5.2	45	90	300	12.30
15	15	5	93	5.2	45	90	400	16.80
16	6	6	93	5.2	45	130	400	16.80

Results and Discussion

Thickness of the electroless Nickel plating was systematically determined using the XRF-based coating thickness measurement.

Normal probability plot

The normal probability plot is as shown in Figure 2. The effects that are plotted along the line are not significant, whereas the main significant effects are away from the line and shown by the boxes placed on the right. The outcomes of this research study arising from the experiments conducted are the following:

i. Bath temperature,

ii. Plating time,

iii. pH, and

iv. Nickel concentration.

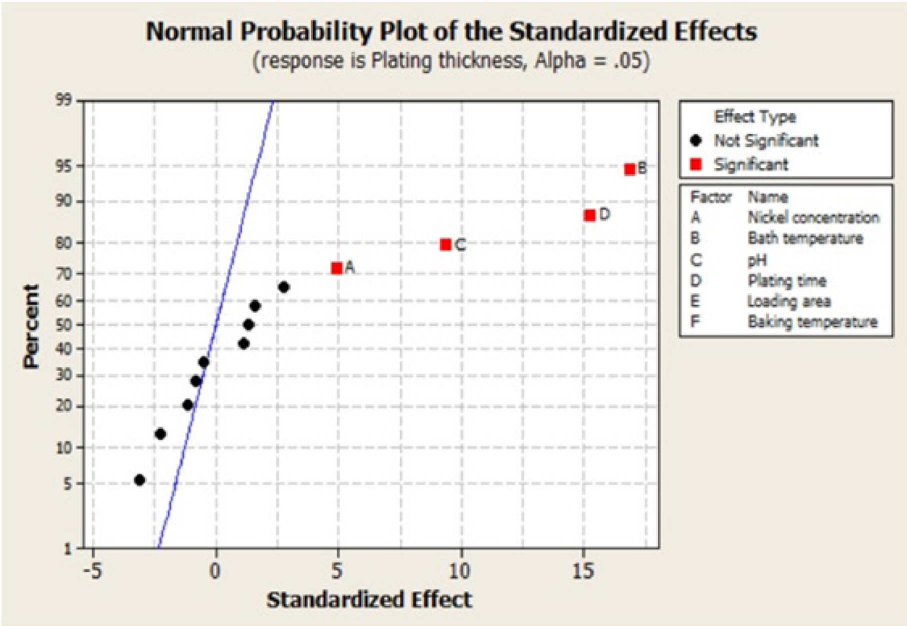

Figure 2: The normal probability plot showing the standardized effects.

Pareto chart

The bars representing factors of bath temperature, plating time and bath pH cross the reference line in this pareto chart, which is 1.456. For the current model, these factors become statistically significant at 0.05 percent.

Main effects plot

The influence of nickel concentration, bath temperature, pH and plating time have a positive trend. It is possible to run all the four variables at a chosen high level to optimize thickness of the nickel plating. The loading area was observed to have minimal influence on thickness of the nickel-plating. In fact, its effect was negative thereby indicating that to increase thickness of the nickel plating the loading area should be minimal. Standardized effect plays an important role in the process of plating thickness. Among the six parameters considered; a, b, c and d play an important role. That means nickel concentration, pH, plating time and bath temperature can be safely considered to being both important and essential towards establishing an optimal thickness of the plating (Figures 3 & 4).

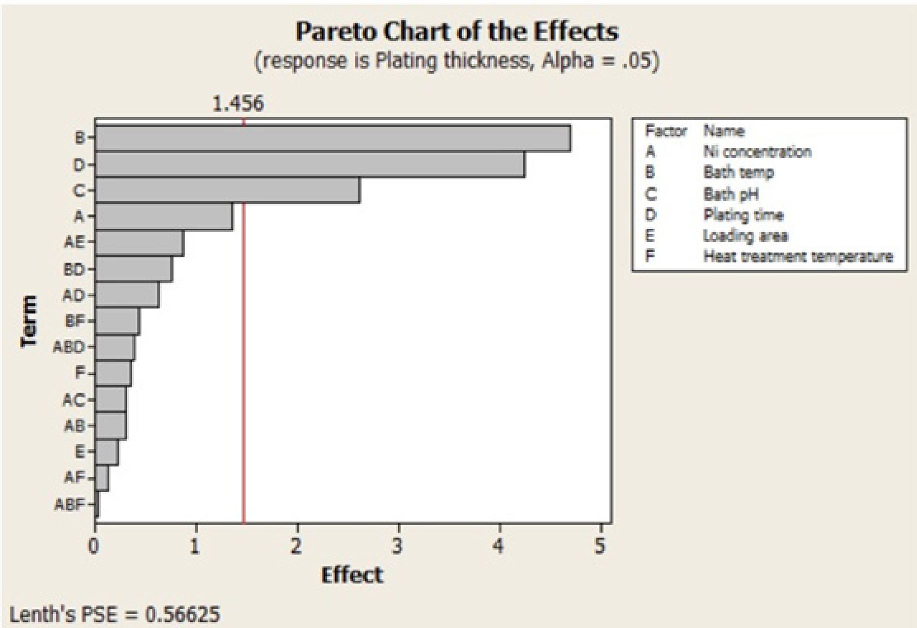

Figure 3: The Pareto chart showing the effects.

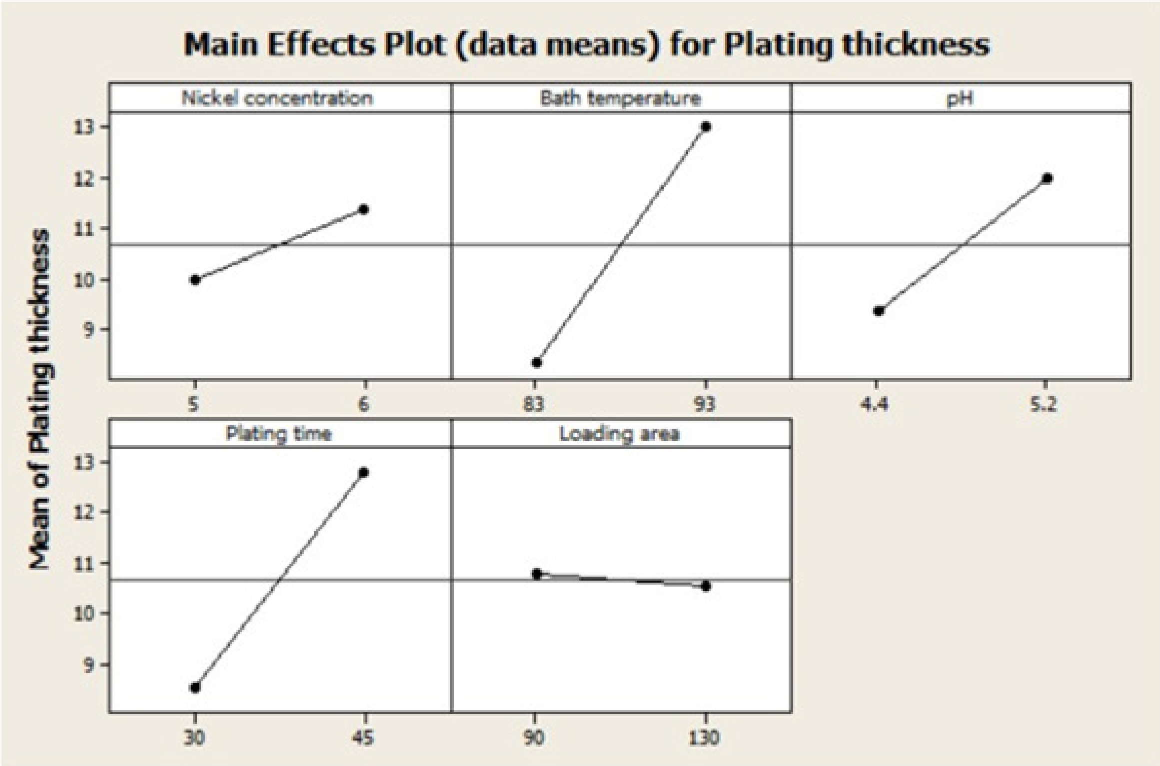

Figure 4: The main effects plot for plating thickness.

Analysis of variance (ANOVA)

The Analysis of Variance [ANOVA] for thickness of the nickel plating was calculated using MINITAB software and adjusted for the tests [9,10]. The details are tabulated in Table 3. From this ANOVA table for the experiments conducted, the 'p' value is low for bath temperature, bath pH and plating time. Hence, the major factors contributing to plating thickness are (i) bath temperature, (ii) bath pH, and (iii) plating time.

Table 3: ANOVA table for the chosen factors.

Degree of Freedom	Sequential Sum of Squares	Adjusted Sum of Squares	Adjusted Mean Squares	Value of Variance Distribution [Obtained after Statistical Experiment]	Probability	
Nickel Concentration	1	7.439	7.439	7.439	7.27	0.025
Bath Temperature	1	88.313	88.313	88.313	86.35	0
Bath pH	1	27.431	27.431	27.431	26.82	0.001
Plating time	1	72.463	72.463	72.463	70.86	0
Loading area	1	0.214	0.214	0.214	0.21	0.658
Heat treatment temperature	1	0.522	0.522	0.522	0.51	0.493
Error	9	9.204	9.204	1.023		
Total	15	205.586				

Interaction plot

From an interaction plot for the plating thickness, we observe that all lines are parallel with no interactions occurring between the chosen factors (Table 4). The figure depicts the extent of correlation between two factors especially when the factors are interacting (interdependent). In the graph shown all the factors selected except loading area and nickel concentration are slightly indirect (Figure 5). All other factors do exert or have a direct correlation with thickness of the plating

Table 4: ANOVA table for main factors and interaction effects.

Source	Degree of Freedom	Sequential Sum of Squares	Variance Value (F)	Probability (P)
Bath Temperature	1	88.313	21.32	0.112
Bath pH	1	27.431	10.56	0.28
Plating time	1	72.463	38.63	0.225
Bath Temperature * Bath pH	1	3.054	2.45	0.152
Bath temperature * Plating time	1	2.333	1.87	0.204
Bath pH * Plating time	1	0.788	0.63	0.447
Error	9	11.205		
Total	15	205.586		

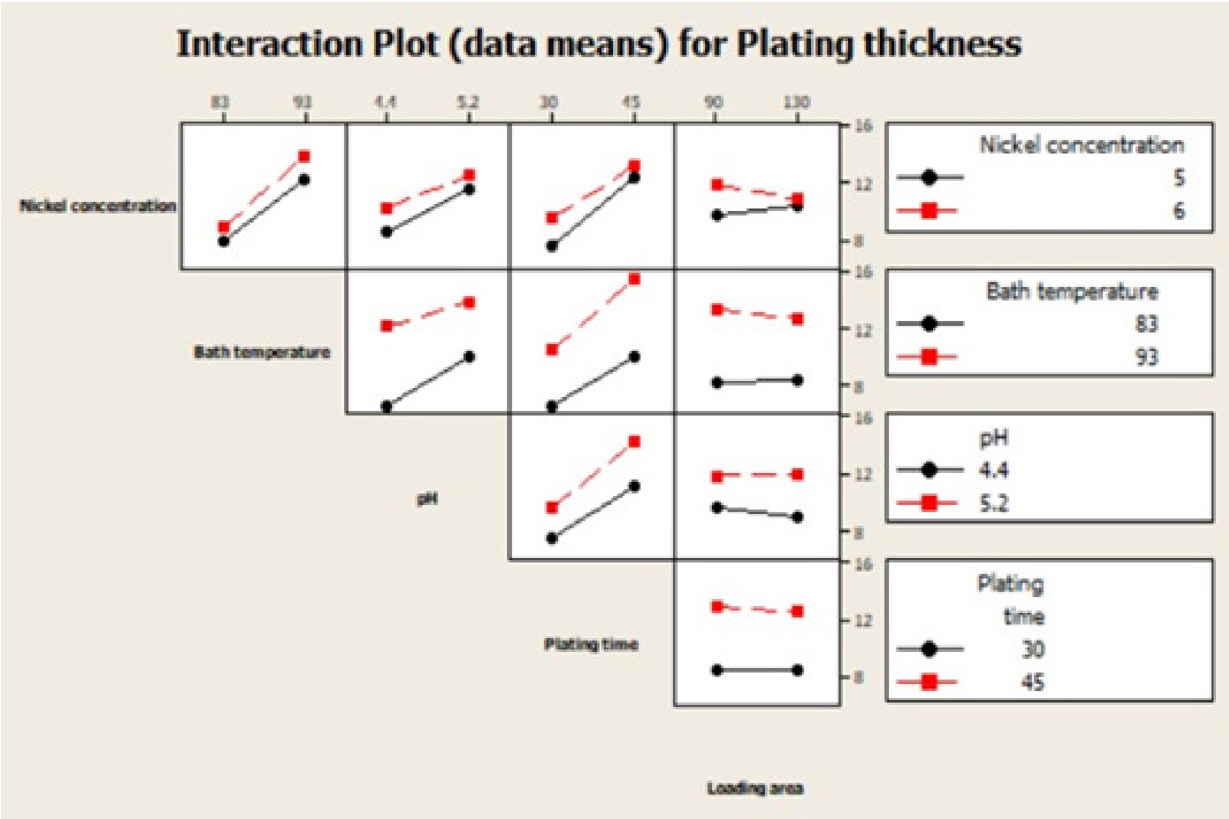

Figure 5: Interaction plot for plating thickness.

Conclusion

(a) The current study brings to light the importance of design of experiment approach in the electroless plating of nickel.

(b) By using fractional factorial design and by varying the six key bath parameters, the experiments were conducted. Thickness of the components was determined using a spectrometer.

(c) The results were carefully analysed using MINITAB software, to establish the analysis of variance (ANOVA), normal probability, main effect and the interaction effects.

(d) From the results the main factors that exert an influence on thickness of the nickel plating are (i) bath temperature, (ii) bath pH, and (iii) plating time.

(e) In order to obtain a specified thickness of the electroless nickel plating the corresponding values of the bath parameters can be obtained from the contour plots.

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