Statistical Analysis on The Effect of Process Parameters on The Thickness of Electrodeposited Co-Fe-Ni Alloy Coating

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ABSTRACT

This research study is a set of experiments based on Co-Fe-Ni alloy coating acquired through the electrodeposition process. Due to its strong magnetic properties, the alloy coating has been applied as a protective layer for metals, particularly in microelectromechanical systems (MEMS). The main focus is on the thickness of Co-Fe-Ni coatings which has an impact on their durability to sustain their function. The combined influence of current density, temperature, and pH value on the thickness of the electrodeposited Co-Fe-Ni coating was studied using an orthogonal central composite experimental design. As a result, the ideal range of the three variables involved is determined in order to get the required properties for the Co-Fe-Ni coating by using the Minitab software. The findings of previous works utilising the Response Surface Methodology (RSM) are analysed using the Minitab programme to create a comprehensive study. The graphical representation was also used to identify the response surface of Co-Fe-Ni electroplating in order to identify the interaction effect, which comprised of a 2D contour plot and a 3D surface plot. According to the findings, obtaining reasonable thickness values for electrodeposited Co-Fe-Ni alloy needs a low current density within the range 0.5–0.8 A/cm^2 and a high temperature bath between 52–54°C. In this case, the quality of the Co-Fe-Ni electroplating deposit has been estimated to be the best based on experimental findings.

Keywords: *Co-Fe-Ni Coating, Electrodeposition, Thickness, Response Surface Methodology*

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Introduction

Mild steel is a kind of carbon steel with a low carbon content, often known as "low carbon steel". Mild steel has a carbon content of 0.05% to 0.25% by weight, while higher carbon steels have a carbon content ranging from 0.30% to 2.0% [1]. Exceptional technologies and analytical methods are being created in many engineering fields to work on the surface properties of metallic materials utilized in industries. Significant efforts to study on the thickness, hardness, corrosion and wear resistance of alloy coatings on mild steel have been broadly explored. It was accounted that Co-Fe-Ni has improved the life span and performance of steel, since its coatings provide an effective and conservative method of securing steel against corrosion [2]. Co-Fe-Ni will be utilized as a coating material for the coating of mild steel in this project. The application of ultra-fine grain size alloy coatings, such as Co-Fe-Ni, has improved chemical and physical characteristics. Co-Fe-Ni alloys, in particular, are recognised for their high Curie temperature and low coercivity, which make them ideal for actuators, step motors, magnetic sensors and hard disk drives. It is likely that a collection and overview of previous work of this alloy coating may be useful in facilitating future study. The findings may be utilised to model and optimise the factors involved in the electrodeposition process in order to achieve the optimum thickness values.

Electroplating or electrodeposition is a process of electrochemically covering the surfaces of a metal article with a layer of another metal [3]. Electrodeposition is a well-known process for producing in situ metallic coatings by passing an electric current through a conductive material submerged in a solution containing a salt of the metal to be deposited [4]. Research studies stated that during the electrodeposition process, the characteristics of the Co-Fe-Ni alloy coating were influenced by an external magnetic field [5]. In this study, the research is restricting the options toward the statistical analysis on the process parameter on the thickness associated with electrodeposited coating.

The research will be done by utilizing the Minitab programming. A well-designed experiment can provide statistical data analysis with a high level of certainty and clarity about the outcome. Response surface methodology (RSM) is used to study the relationship between a large number of explanatory variables and one or more response variables. RSM uses a series of well-prepared tests to obtain an optimum response [6].

The Design of Experiment (DOE) method is used to restrict the number of component trials by attributing a high and low value to each variable when a large number of variables, also known as factors, are involved and the experiment requires them to be monitored at the same time [7]. Design of experiment is a type of statistical analysis used in the scientific and engineering fields. These studies have shown considerable benefits in allowing researchers and engineers to analyse their data from a variety of studies in a systematic and orderly manner [8]. The factorial design is one of the options, and it may be enhanced by using the central composite design, which yields more exact results. This design is relevant because it is frequently used in research and may be used to create new designs with practical utility [9].

Methodology

The current density, J (A/dm^2), temperature, T (°C) of the bath during the electroplating process, and pH value of the electrolyte solution were the parameters employed in this study for the response surface approach. As a result, the parameter values were obtained and presented in Table 1. These characteristics were utilised to generate a numerical study of the deposited Co-Fe-Ni alloy coating, which focused on the thickness of the metallic layer value or responses. The results of the investigation were derived from studies that were carried out in a similar way to how the experiment was carried out. A Positector 6000 non-destructive testing physical approach was used to determine the thickness of the coating layer.

Table 1: Design variables and actual values used for electroplating process

Current density, J (A/dm^2)	1.56 – 3.70
Temperature, T (°C)	24 - 36
Solution pH	2.39 – 3.61

The DOE approach was used to restrict the number of component experiments when a large number of variables or factors are involved and the experiment requires them to be controlled [7]. This approach, according to previous research, is used to limit the number of component tests when the DOE method incorporates a high number of variables called factors and the experiment requires them to be controlled [10]. The finding of variable settings for which the mean response is optimum, as well as the estimation of the response surface in the region of this ideal location, are the purposes of response surface methodology [11]. A 2^3 orthogonal central composite experimental design was employed for the investigation of the response surface methodology (RSM) in a previous study, which used the same sort of design but a different substrate [12]. To reach the best possible result, RSM employs a series of well-designed tests.

According to the experimental design, a total of N = 16 trials were obtained from various previous investigations. An orthogonal central composite design of experiments has been planned to explore the electroplating process for the deposition of Nickel alloys using the coded levels of variables

[13]. The relationship between a number of explanatory variables and one or more response variables is investigated using RSM [12]. A 2^3 orthogonal central composite design with 5 levels (-, -1, 0, +1, +) was used for each independent variable. Throughout the electroplating process, the variables were X_1 - current density, J (A/dm^2), X_2- bath temperature, T (°C), and X_3 - electrolyte pH solution. Two responses were determined experimentally in line with defined runs in order to establish the performance of the Co-Fe-Ni electroplating process. Table 2 shows the orthogonal central composite design with the coded values presented alongside the natural values. These numbers were entered into the Minitab software, where the Minitab software calculated the response surface analysis.

Table 2: Experimental responses were found using a central composite orthogonal design that was applied to organise the electroplating method

Run	Factors (controllable input variables)						Responses	
	Current density		Temperature		Solution pH		Thickness (μm)	Current efficiency (%)
	X_1	J (A/dm^2)	X_2	T (°C)	X_3	pH		
1.	+1	3.51	+1	35	+	3.50	59.125	88.784
2.	-1	1.75	+1	35	+	3.50	17.750	91.171
3.	+1	3.51	-1	25	+	3.50	71.125	80.732
4.	-1	1.75	-1	25	+	3.50	50.625	90.532
5.	+1	3.51	+1	35	-	2.50	14.625	89.116
6.	-1	1.75	+1	35	-	2.50	15.625	80.853
7.	+1	3.51	-1	25	-	2.50	68.000	82.480
8.	-1	1.75	-1	25	-	2.50	16.000	64.617
9.	+α	3.79	0	30	0	3.00	58.375	85.689
10.	-α	1.56	0	30	0	3.00	28.500	86.251
11.	0	2.63	+α	36	0	3.00	16.500	92.317
12.	0	2.63	-α	24	0	3.00	70.000	82.023
13.	0	2.63	0	30	+α	3.61	75.375	90.731
14.	0	2.63	0	30	-α	2.39	22.500	68.394
15.	0	2.63	0	30	0	3.00	46.875	84.932
16.	0	2.63	0	30	0	3.00	45.801	84.851

Results and Discussion

Minitab software has computed all of the parameters in the analysis, including all of the linearity, quadratic relationships and interaction terms. The coefficient value, probability value (p-value), and r-squared (R-sq) are the values mentioned in Table 3 to 6. While the other terms are maintained constant, the coefficient for this term shows the change in the mean response associated with an increase of one coded unit in that term. Thus, the direction of the correlation between the term and the answer is indicated by the sign of the coefficient, which might be positive or negative. The value of the coefficient is half that of the effect, however this does not indicate whether a term is statistically significant because significance calculations take reaction data fluctuation into consideration.

The R-squared values of 85.20% (Table 4) and 89.31% (Table 6) are sufficient to confirm that the model may be used to anticipate future experiment results (thickness of the coating layer and plating current efficiency). The R-squared number is always in the range of 0% to 100%, with 0% indicating that the model explains no variability and 100% indicating that the model explains all variance in the response around its mean. This R-sq value of 85.20% and 89.31% is good enough to show that the factors in this analysis are well generated to the response in this situation, as the values of the variables and response were gathered from various researches. As a result, it can be applied in future studies.

Table 3: Response Surface Regression on the effect of Thickness from Minitab software

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	48.86	7.80	6.26	0.001	
Current density, J	16.67	5.34	3.12	0.021	1.01
Temperature, T	-18.21	5.12	-3.55	0.012	1.00
pH solution	16.63	5.19	3.21	0.018	1.00
Current density, J * Current density, J	-6.63	9.82	-0.67	0.525	1.02
Temperature, T * Temperature, T	-6.11	9.37	-0.65	0.538	1.02
pH solution * pH solution	-0.39	9.53	-0.04	0.969	1.02
Current density, J * Temperature, T	-6.11	7.56	-0.81	0.450	1.00
Current density, J * pH solution	2.10	7.68	0.27	0.794	1.00
Temperature, T * pH solution	1.62	7.28	0.22	0.831	1.00

Table 4: Model summary of the design of Thickness from Minitab

S	R-sq	R-sq(adj)	R-sq(pred)
14.0611	85.20%	63.01%	0.00%

Table 5: Response Surface Regression on the effect of Current Efficiency from Minitab software

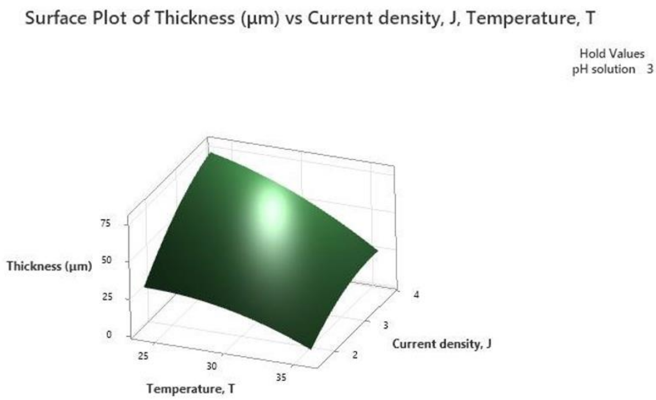
Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	85.00	2.24	37.99	0.000	
Current density, J	1.55	1.53	1.01	0.352	1.01
Temperature, T	4.83	1.47	3.28	0.017	1.00
pH solution	6.53	1.49	4.39	0.005	1.00
Current density, J * Current density, J	0.88	2.82	0.31	0.766	1.02
Temperature, T * Temperature, T	2.29	2.69	0.85	0.426	1.02
pH solution * pH solution	-5.32	2.73	-1.95	0.100	1.02
Current density, J * Temperature, T	-0.42	2.17	-0.19	0.854	1.00
Current density, J * pH solution	-7.40	2.20	-3.36	0.015	1.00
Temperature, T * pH solution	-2.60	2.09	-1.24	0.260	1.00

Table 6: Model summary of the design of Current Efficiency from Minitab

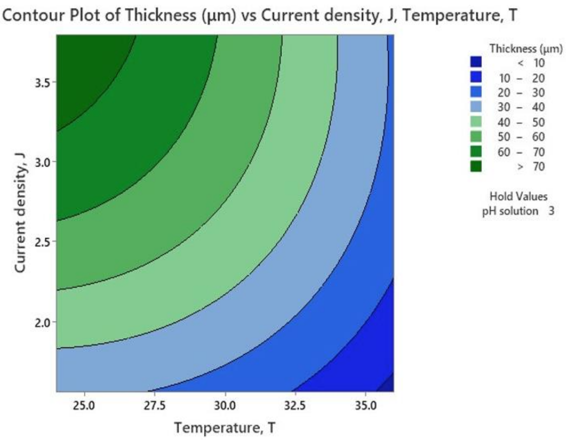
S	R-sq	R-sq(adj)	R-sq(pred)
4.03333	89.31%	73.28%	0.00%

Figure 1 illustrates the response surface plots and contour areas for thickness, current density, and temperature in relation to three variables. It is indeed worth mentioning that for the single response surface plot, only two elements can be plotted together against the response axis. In order to examine the response surface, one of the three elements or variables in this analysis must be kept constant. The pH level is kept constant at 3 in this circumstance. On the other hand, contour plots for thickness that take current density and temperature into factor use the same procedure as response plots. Essentially, the contour plot is a two-dimensional version of the response surface plot. The contour plot makes it easy to study and explain the impacts of any two components. The dark green patches in both figures indicate the optimum thickness of more than 70 μm.

The contour plot shows that the highest thickness values may be attained with a lower current density and a moderate temperature. Based on both figures, the current density and temperature range may be computed. The optimum thickness value (> 70μm) for the Co-Fe-Ni alloy coating may be obtained using a current density range of less than 2.0 A/dm^2 and temperature below 27.5°C.



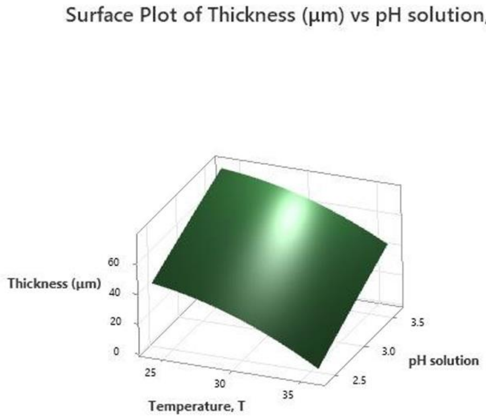
(a)



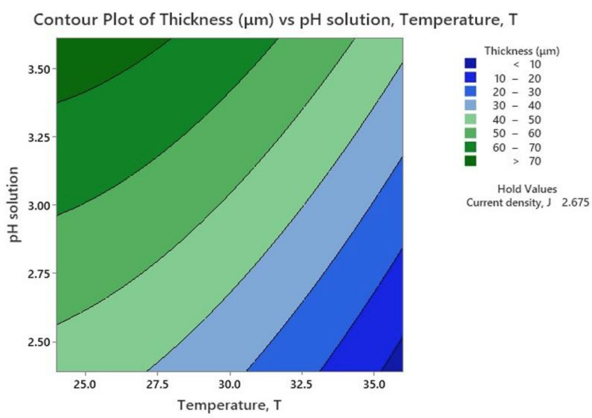
(b)

Figure 1: (a) Surface and (b) contour response plot for thickness against current density and temperature

The response surface plots and contour areas plotted in the context of response for temperature and pH solution are shown in Figure 2. The current density with a value of 2.675 A/dm^2, on the other hand was kept constant. By examining both of these plots, the optimum thickness value is found in the dark green zone, which is stated in the graph's legend as more than 70 µm. As a result, the temperature range for achieving the optimum thickness value is lower than 27.5°C, with a pH solution higher than 3.3. The highest thickness value area may be observed towards the upper left corner of the contour plot. A higher pH and a suitable temperature value will also produce a higher thickness value for the alloy coating layer.



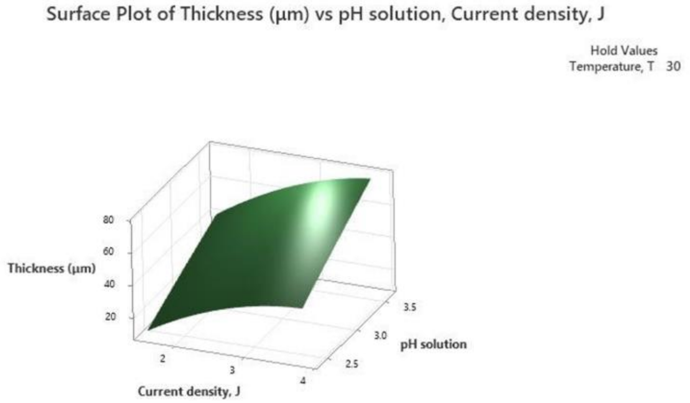
(a)



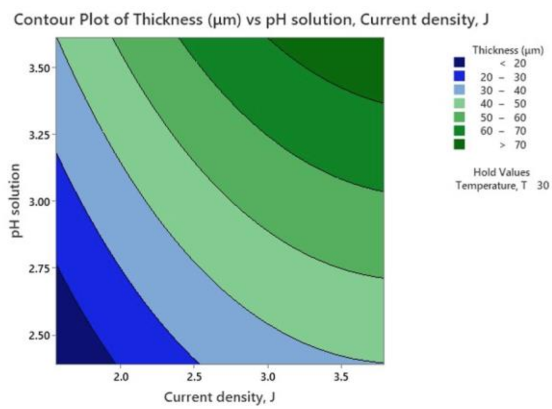
(b)

Figure 2: (a) Surface and (b) contour response plot for thickness against pH solution and temperature

Figure 3 shows the response surface plots and contour areas plotted in the context of response for factors of pH solution and current density. Meanwhile, the temperature of 30°C was maintained as a constant. By examining both of these plots, the optimum thickness value is found in the dark green region, which is shown in the legend of the graph as thicker than 70 µm. From this, it can be stated that the pH solution should be more than 3.0 to produce the highest thickness value. A higher pH value may also be claimed to produce better thickness, although the current density value has no significant influence on the thickness of the metallic layer.



(a)



(b)

Figure 3: (a) Surface and (b) contour response plot for thickness against pH solution and current density

Due to its excellent quality as a head core material in hard disk drives, Co-Fe-Ni alloy is among the most investigated soft magnetic materials. Due to hydrogen plating, conventional low pH (2.5–3.0) baths have problems such as poor stability, low current density efficiency and voids in the deposited film. Citrate may significantly improve the stability of Co-Fe-Ni plating baths, and because of the higher pH of the bath (> 5), a denser Co-Fe-Ni film can be obtained [15]. Different potentials and the associated additive coverage of the Co-Fe-Ni surface are established depending on the magnitude of the pulse currents utilised, impacting the Co-Fe-Ni alloy composition, crystal structure, magnetic characteristics, and deposit surface quality [4].

Conclusion

The Minitab programme was used to create an orthogonal composite central design using the current density, temperature, and pH solution as the response variables. Overall, the maximum thickness values for the Co-Fe-Ni alloy coating were obtained with an average current density, average electrodeposition bath temperature, and a high pH value, according to the results analysis. The thickness of metallic coating layer response of electrodeposited Co-Fe-Ni alloy coating was investigated and analysed by using the design of experiments (DOE) method and the modelling and optimisation process was conducted using the response surface methodology (RSM). From the analysis done is was found that the optimum thickness of about 70 μm could be achieved by using higher pH value of 3.3. Low current density and bath temperature was found to also help in achieving a thick and dense coating layer.

Other elements in the electrodeposition bath, such as the iron content, the inclusion of organic additives, and other electrodeposition parameters, must also be considered since these factors can impact the alloy coating's thickness values. The design of this model could be used to optimise response values as a valuable tool may be applied to future tests to reduce costs and time spent on conducting several experiments.

The study focused on the response of thickness of Co-Fe-Ni alloy coatings, other responses such as coating strength or stresses, magnetic properties, microstructure grain sizes, and other measurements could be measured to model and improve the alloy coatings. The main purpose of the response surface approach is to find the suitable variables for the optimal outcomes. Furthermore, this method is commonly used prior to doing bulk testing on the coatings. In addition, the same form of analytic methodology used in this research study may be used and adapted to a variety of alloys.

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