OPTIMIZATION OF MECHANICAL BEHAVIOR OF NI-P NANOCOMPOSITE COATINGS USING TAGUCHI APPROACH

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ABSTRACT

This paper studies the electroless (Ni-P) deposition which used in different engineering applications due to their ability to modify and enhance the surface properties of the steel substrate. The electroless plating process was used to prepare (Ni-P/Nano TiO₂), (Ni-P/Nano Al₂O₃) and (Ni-P/Nano SiO₂) alloys in this research. Deposition process parameters based on (L28) Taguchi orthogonal configuration with three process parameters, viz., stirring speed, temperature, time, are designed for optimum microhardness. Under the Taguchi series, the microhardness activity of electroless (Ni-P-TiO₂) nanocomposite deposition was measured. The findings revealed that the integration of TiO₂ into the coating allows micro-hardness to increase. Finally, optimum conditions were achieved as, A2B1C2 (i.e. middle-level of bath temperature (90°C), lower level of plating time (80 min.) and middle level of nanomaterial (Al₂O₃)), the ANOVA analysis reveals that nanomaterial has a great (60%) influence over the microhardness and surface roughness as compared to bath temperature (28%) and plating time (12%).

KEYWORDS: Nanocomposite Coatings, Mechanical properties, Taguchi Method

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1.INTRODUCTION

Generally, deposition of Electroless nickel-phosphorus has been examined because it became to start with defined through Brenner and Riddell in 1946 [1]. The electroless Ni-P alloys are usually utilized in different engineering applications for their typical mechanical and physical properties such as excellent wear, abrasion and corrosion resistance, ductility, soldering, lubrication, and electric properties in addition to low cost of material [2-3]. Among the electro-plating techniques, electroless deposition has more advantages because of its low device cost, easy in operation, and the formation capacity of very thin and uniform coating on each conductive and non-conductive surfaces. [4-5]. Electroless plating is an autocatalytic method. In this process the reduction of the metal ions and coating deposition may be done via the oxidation of the lowering agent, which materials an electric powered. The substrate develops ability while it is dipped in electroless solution known as electroless bath, which includes a supply of metal ions, reducing agent, complexing agent, stabilizer, additives, and wetting agents etc. Due to the advanced ability, each positive and negative ions are attracted in the direction of the substrate surface and release their energy via charge transfer method The prime characteristic of electroless nickel-phosphorus coating is the uniform deposition of coating on metal (ferrous and non-ferrous) substrate. Electroless coatings can widely be classified into four categories viz, pure nickel and black nickel coating, alloy and poly-alloy coatings, composite coatings and electroless nano coatings. Electroless plating has highly consistent thickness across all surfaces, such as edges and complex interior geometry [6]. With current advances in nanoparticles technology and their applications in different fields [7],

also, they were used as an additive in electroless Ni/P plating. Composite coating is ready with the aid of using including solid particles alumina, titanium oxide, silicon carbide, silicon nitride, carbon nanotube into electroless coating solution [8]. These coatings show higher physico-mechanical properties compared with plane Ni/P electroless coatings. One of the prime properties which has proved to be of significance in successful applications is hardness [9]. Surface hardness is especially essential for the control of tear and wear methods in components or products. The hardness of the electroless deposition will increase with the heat treatment to a certain temperature [10]. The microhardness of the surface has a extensive effect on mechanical properties including fatigue behavior, resistance to corrosion and creep life. As a result, development in the modeling of surface microhardness and optimization of control parameters is required to achieve the desired degree of surface microhardness. It is therefore necessary to maximize the mean microhardness of the products [11-12]. In composite coatings surface roughness performs very essential role because it influences on porosity of coating. Due to higher porosity the corrosion resistance of the composite coating can be reduced. Hence it is important to reduce or control the surface roughness of coated samples. In this research, the Taguchi technique become used for optimization [13-14]. The aim of this research is to improve the mechanical properties of the low carbon steel substrate by coating it electrolessly with Ni-P-TiO₂, Ni-P-Al₂O₃ and Ni-P- SiO₂ coating and find the optimal coating conditions.

2. EXPERIMENTAL

2.1. Substrate deposition and preparation:

Low carbon steel samples with the dimensions (1.5 cm × 1.5 cm × 0.5 cm) were used as substrates. The chemical structure of the low carbon steel shows in the table (1) by using Spectra Max at (The General Company for Engineering Inspection and Rehabilitation):

Table 1: Chemical composition of Low carbon steel

Element	C	Si	Mo	S	P	Ni	Mn	Al	Cr	Pb	Fe
Wt%	0.139	0.0072	0.002	0.0053	0.0088	0.0324	0.526	0.0522	0.0044	0.0030	Bal

Then specimens washed at room temperature in an alkaline solution (1 M KOH) after which successively rinsed with suitable deionized waters for 15 minutes. Before the deposition, the steel sample cleaning is accomplished as follows The samples had been mechanically polished to (2000) grade with emery papers, and carefully processed at a (50%) dilute (HCl) solution. Specimens had been washed with water, dried manually through a piece of cotton after which washed through methanol. Samples after washing with methanol had been rinsed and dried in an oven. The samples then had been immersed in the (Ni-P) deposition bath instantly. Table (2) shows the tank composition for the (Ni-P) Nano (TiO₂) electroless deposition. The tank is run at (80 °C to 100 °C) with a (pH = 5) solution for (40 minutes to 120 minutes) of plating time.

Table 2: Bath composition for (Ni-P) Nano (TiO₂) deposition.

Substance	Concentration (g/L)
NiSO ₄ ·6H ₂ O	30
NaH ₂ PO ₂	25

Na ₃ C ₆ H ₅ O ₇ . H ₂ O	20
Thiourea	0.002
Nano TiO ₂	3

Table (3) shows the bath of composition for (Ni-P) Nano (Al₂O₃) electroless deposition. The bath was worked at (80 °C to 100 °C) with a (pH = 5) solution for (40 minutes to 120 minutes) of plating time.

Table 3: Bath composition for (Ni-P) Nano (Al₂O₃) deposition.

Substance	Concentration (g/L)
NiSO ₄ ·6H ₂ O	30
NaH ₂ PO ₂	25
Na ₃ C ₆ H ₅ O ₇ . H ₂ O	20
Thiourea	0.002
Nano Al ₂ O ₃	3

Table (4) shows the bath of composition for (Ni-P) Nano (SiO₂) electroless deposition. The bath was worked at (80 °C to 100 °C) with a (pH = 5) solution for (40 minutes to 120 minutes) of plating time.

Table 4: Bath composition for (Ni-P) Nano (SiO₂) deposition

Substance	Concentration (g/L)
NiSO ₄ ·6H ₂ O	30
NaH ₂ PO ₂	25
Na ₃ C ₆ H ₅ O ₇ . H ₂ O	20
Thiourea	0.002
Nano SiO ₂	3

In each experiment, (200 mL) of fresh bath solution was selected.

2.2 Microhardness measurements

For evaluating the microhardness of electroless (Ni-P) Nano (TiO₂), (Ni-P) Nano (Al₂O₃) and (Ni-P) Nano (SiO₂) coatings, a microhardness tester with a Vickers indenter was used. To cause the indentations in all deposits, a constant load of 100 g was applied and the hardness values were averaged out of three such determination. The test was done in (Department of Materials Engineering - University of Technology - Baghdad - Iraq), as shown in figure (1):

Figure 1: Microhardness test device.

2.3 Taguchi Method

Design of Experiments (DOE) is based on the objective of desensitizing a product's performance characteristic(s) to variation in critical product and process design parameters. Through DOE, a series of tests are performed where preplanned changes are made to the controllable variables so that the reason for changes in the response can be observed and identified. When DOE is performed using Taguchi method, the latter generally relates the variability in the responses of a particular trial condition with the effect of the uncontrollable variables or noise. Taguchi method obtains the optimal condition by observing the reduction in variation of the results within a particular trial condition. Hence, to take into account the variation of results, Taguchi method uses the S/N ratio to identify the quality characteristics applied for engineering design problems. In the S/N ratio, signal is interpreted as mean while noise is interpreted as standard deviation.

It is found that the characteristics of electroless coating get affected by several factors such as bath temperature, plating time, coating composition using nanomaterial, stabilizer concentration, pH of the solution, substrate, bath load, etc. Consideration of all the factors would make the experimental design highly complex and the analysis even more complicated. A review of the recent literatures revealed that the three factors viz. bath temperature (A), plating time (B) and coating composition using nanomaterials (C) are the mostly preferred factors used by the researchers to control the properties of electroless nickel deposits. The bath temperature initiates the reaction mechanism and thereby determines the rate of reaction by controlling the ionization and charge transfer process. The considered design parameters, together with their levels are shown in Table 5. Consideration of three levels allows the study of non-linear effects if any.

Table 5: Design parameters and their levels

Design Factors	Unit	Levels		
		1	2	3
Bath temperature A	°C	80	90	100
Plating time B	Min.	40	80	120
Nano-material C	-	TiO ₂	Al ₂ O ₃	SiO ₂

In the present article, microhardness and roughness of electroless Ni-P coatings were studied. Hence, the two popularly evaluated parameters are considered as the response variables. A lower value of *roughness* and a higher value of *hardness* indicate that the material under test has a higher resistance against abrasive.

In the context of DOE, the number of degrees of freedom of a particular parameter is one less than the number of levels associated with the parameter. In the present case since each of the main factors is associated with three levels, the dof of each of the factors is two. It is important to notice that the number of experimental trials in the Orthogonal Array OA must be greater than the total dof required for studying the effects. Hence, L_{27} OA, requiring twentyseven experimental runs is suitably chosen for the present case. The assignment of the factors to the columns of the array is done on the basis of the Triangular Table for 3-level OA. The L_{27} OA together with the column assignments are shown in Table 6. The values in each cell of the main parameter columns (A, B and C) in the array indicate their levels (1, 2 and 3).

Table 6: L_{27} orthogonal array with main parameters

Exp.No.	Nano material	Temperature	Time	Speed of stirring
1	Tio2	80	40	500
2	Tio2	80	80	500
3	Tio2	80	120	500
4	Tio2	90	40	500
5	Tio2	90	80	500
6	Tio2	90	120	500
7	Tio2	100	40	500
8	Tio2	100	80	500

9	Tio2	100	120	500
10	Al2o3	80	40	500
11	Al2o3	80	80	500
12	Al2o3	80	120	500
13	Al2o3	90	40	500
14	Al2o3	90	80	500
15	Al2o3	90	120	500
16	Al2o3	100	40	500
17	Al2o3	100	80	500
18	Al2o3	100	120	500
19	Sio2	80	40	500
20	Sio2	80	80	500
21	Sio2	80	120	500
22	Sio2	90	40	500
23	Sio2	90	80	500
24	Sio2	90	120	500
25	Sio2	100	40	500
26	Sio2	100	80	500
27	Sio2	100	120	500

3. RESULTS AND DISCUSSIONS

Taguchi method needs to take into account the variability within a trial condition. The variability can be easily captured if S/N ratio is used to convert the experimental results into a value for the evaluation characteristic in the optimum parameter

analysis, instead of the mean. The idea is to maximize the S/N ratio, thereby minimizing the effect of random noise factors, which have a significant impact on the process performance. In the present work S/N ratio analysis is done with microhardness and roughness as the performance index and all the calculations are conducted in Minitab, where microhardness is to be maximized and roughness is to be minimized, S/N ratio.

The microhardness and roughness values obtained from experimentation along with their S/N ratios is shown in Table 7.

Table 7: Experimental results along with S/N ratio

Exp.No.	Nano material	Temperature	Time	Speed of stirring	Microhardness	Roughness	S/N ratio
1	Tio2	80	40	500	201.4	0.9395	-2.92926
2	Tio2	80	80	500	265.8	0.7195	-2.96328
3	Tio2	80	120	500	274.3666667	0.9695	-2.94891
4	Tio2	90	40	500	221.3333333	1.3335	-2.90564
5	Tio2	90	80	500	417.8666667	0.748	-2.9792
6	Tio2	90	120	500	254.5	1.6805	-2.89559
7	Tio2	100	40	500	340.1333333	1.474	-2.93502
8	Tio2	100	80	500	164.2	0.533	-2.95391
9	Tio2	100	120	500	341.2	1.645	-2.92655
10	Al2o3	80	40	500	128.0666667	0.6735	-2.91894
11	Al2o3	80	80	500	248.4333333	1.3735	-2.91426
12	Al2o3	80	120	500	321.0333333	0.826	-2.9656
13	Al2o3	90	40	500	354.5666667	1.4055	-2.94144
14	Al2o3	90	80	500	316.2	1.398	-2.93349
15	Al2o3	90	120	500	352.5	1.103	-2.95594
16	Al2o3	100	40	500	237.3333333	1.176	-2.92422
17	Al2o3	100	80	500	473.8	0.7485	-2.98286
18	Al2o3	100	120	500	394.0333333	0.8605	-2.97236
19	Sio2	80	40	500	141.7333333	0.765	-2.91654
20	Sio2	80	80	500	143.3333333	0.099	-2.9983
21	Sio2	80	120	500	138.1333333	0.373	-2.96339

22	Sio2	90	40	500	132.6666667	0.5155	-2.9428
23	Sio2	90	80	500	299.1666667	0.513	-2.98051
24	Sio2	90	120	500	509.5666667	1.063	-2.97406
25	Sio2	100	40	500	136.3666667	0.7355	-2.9166
26	Sio2	100	80	500	168.1	1.182	-2.88815
27	Sio2	100	120	500	516.2666667	0.6315	-2.98905

As it is known that the columns of the OA are orthogonal to each other, the average effect of each factor on the quality characteristic at different levels can be determined. The average of the S/N ratio for each level of the factors of A, B, and C are given in Table 8. The delta value was calculated by subtracting the largest value from the lowest from among the values in each column.

Table 8: Response Table for Signal to Noise Ratios

Level	Nano material	Temperature	Time
1	-2.945	-2.946	-2.926
2	-2.952	-2.945	-2.955
3	-2.937	-2.943	-2.955
Delta	0.015	0.003	0.029
Rank	2	3	1

Very simply, a design factor with a large difference in the signal noise ratio from one factor setting to another indicates that the factor or design parameter is a significant contributor to the achievement of the performance characteristic. When there is little difference in the signal to noise ratio from one factor setting to another, this indicates that the factor is insignificant with respect to the performance characteristic. It is found from Table 8 that plating time the highest delta value and hence has the greatest influence over the wear performance of electroless Ni-P coatings. The main effect plot are illustrated in Fig.2. The main effects plot gives the optimal combination of testing parameters for minimum roughness and maximum microhardnes.

Figure 2: Main effects plot for signal to noise ratio

Since Taguchi method obtains the optimal level combination by choosing those levels for which S/N ratio is the highest, the optimal combination of parameters is found to be A2B1C2 i.e. middle-level of bath temperature (90°C), lower level of plating time (80 min.) and middle level of nanomaterial (Al₂O₃). Moreover, the main effect plot also gives a rough idea about the relative significance of the parameters on the system response. This is determined by the slope of the main effect plot for each parameter. The plot having higher inclination will have higher influence.

ANOVA is a statistical technique to find out the significance of individual process parameters and their interactions on the system response under consideration. In the present study, ANOVA is applied with an objective to evaluate the significance of testing parameters on the microhardness and Roughness Performance of electroless Ni-P coatings. If some testing parameters do not have considerable impact on microhardness and Roughness Performance, they can be kept within a suitable range for the test and can be excluded in building future prediction and optimization models. The percentage contribution of variance can also be calculated through ANOVA. In the present study, ANOVA is performed using S/N ratio as the response and the results are shown in Table 9.

Table 9: Results of ANOVA

Source	DF	SS	MS	% Contr.
Nano material	2	1.1544	0.5772	60
Temperature	2	0.5474	0.2737	28
Time	2	0.2332	0.1166	12

Error	20	2.2582	0.1129	
Total	26	4.1932		

ANOVA table shows the percentage contribution of each parameter. It is seen from the table that the parameter A (Nanomaterial) has got the most significant influence on microhardness and surface roughness at confident level of 95% within the specific test range as compared with the bath temperature (12%) and plating time (12%).

The final step of the DOE is the confirmation test, which verifies if the optimum conditions suggested by the matrix experiment do indeed give the improvement projected. The verification experiment is performed by conducting a test with optimal settings of the factors and levels previously evaluated. The predicted value of the S/N ratio at the optimum level η is calculated as:

$$\eta = \eta_m + \sum_{i=0}^o (\eta_i - \eta_m) (1 - 1)$$

where η_m is the total mean S/N ratio, η_i is the mean S/N ratio at the optimal level, and o is the number of main design parameters that significantly affect the wear performance of electroless Ni-P coating. Table 10 shows the comparison of the predicted S/N ratio with the actual (experimental) S/N ratio using the optimal parameters and there seems to be quite a good agreement between the two.

Table 10: Results of the confirmation experiment

	Initial Parameters	Optimal Parameters	Experiment
Level	A1B1C1	A2B1C2	A2B1C2
Microhardness & Roughness	-2.92926	-2.96347	-3.1321

Figure 3: X-ray diffraction of electroless Ni-P-Al₂O₃ Coating

4. CONCLUSIONS

In this study Taguchi method, is successfully applied in order to optimize the electroless characteristics of Ni-P coatings. Accordingly, the following results were obtained:

- Taguchi method could be a suitable DOE for Ni-P nanocomposite coatings.
- Taguchi method showed that nanomaterial had the highest impact on the microhardness and surface roughness of Ni-P nanocomposite coatings.
- Optimal parameters for the formation of Ni-P nanocomposite coatings included A2B1C2 i.e. middle-level of bath temperature (90°C), lower level of plating time (80 min.) and middle level of nanomaterial (Al₂O₃).
- The ANOVA analysis reveals that nanomaterial has a great influence over the microhardness and surface roughness performance of the coating as compared to bath temperature and plating time.
- The XRD plot revealed that the coating possesses a mixture of amorphous and crystalline structure.

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