

Corrosion Behavior Optimization by Nanocoating Layer for Low Carbon Steel in Acid and Salt Media

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In this paper, a SiC nano electroless nickel plating layer with excellent corrosion resistance was fabricated using the Taguchi method. The electroless plated low carbon steel was subjected to tests to examine the influence of corrosive media, microhardness, and corrosion rate on the corrosion resistance of this alloy. Three different corrosive media (HCl, Na₂SO₄, and NaCl) at various temperatures (80, 90, and 100 °C) were used, and at three different times (40, 80, and 120 min.) with a speed of stirring equal to 500 rpm. The results of microhardness were found from 134.276 HV to 278.578 HV at various conditions, while the corrosion rate results were obtained from 0.89643 mpy to 7.12571 mpy at different circumstances. Corrosion, and mechanical characteristics were explained using Taguchi design. Taguchi technique was used to account for all possible combinations of elements in order to conduct a complete study. Models that link the response and procedure parameters were developed using the results of these tests, and the analysis of variance was utilized to validate these models (ANOVA). For maximum efficiency, a function called “desirability” was applied to all responses at once.

Keywords : *Electroless plating, Corrosion resistance, Nanocomposites coatings, Taguchi method*

1. Introduction

Corrosion is the attrition of metals caused by an attack element(s) in their environment, such as fluorine, chlorine, carbon dioxide, oxygen, and so on [1]. Surface treatment, such as the use of barrier(s) like coatings and films to reduce cracks, can help to prevent the corrosion [2]. Because of the vast variety of coating techniques and coating materials available for use in a diversity of conditions and applications, coating is the most extensively used method of reducing, preventing or controlling the corrosion [3,4]. The coating is a means of completing the surface of an item, which is referred to as the substrate in most circumstances. Coatings are used to improve the mechanical characteristics, adhesion, wettability, corrosion resistance, and wear resistance of the substrate [4]. Nanomaterial(s) have recently been included as a viable means of reducing

corrosion. Nanomaterial(s) are material(s) with at least one morphological property, such as the size of particle, grain size, structure size, and so on, that is on the nanoscale (less than 100 nm) [5]. The structure, shape, and dimensional of nanomaterials includes: nanoparticles, nanotubes, nanowires, and nano rods that are made up of one-dimensional nanotubes, nanowires, and nano rods that are made up of two-dimensional nano platelets, nano sheets, and nano films that are made up of two-dimensional nano platelets, nano sheets, and nano films that are made up of two-dimensional. Among the many properties of nanomaterials [6] are their advanced thermal and mechanical properties as well as their physical and chemical properties, besides their magnetic and electrical properties. As a consequence of their tiny size, this allows for increased volume fractions on the surfaces, resulting in larger contact areas [7], and this is possible. Nanomaterials have the ability to minimize the rate of corrosion of metal substrates by altering the

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surface of the substrate with a coating layer. Electroless nickel coating(s) were widely employed in the mechanical, aerospace and chemical industries because of their oxidation, corrosion resistance, surface hardness and wear resistance [8]. Because of the low-cost device of electroless coating, its simplicity of operation, and its capacity to produce very thin and uniform film coatings on both conductive and non-conductive surfaces, it provides various benefits [9]. Nickel ions are catalytically lowered on active substrates without utilizing electrical energy in an Electroless plating procedure, and the electrons necessary for the reduction reaction are provided by chemical reducing agents [10]. Phosphorus is one of the most common chemical reducing agents, and the resultant is known as Nickel-phosphorous (Ni-P) plating. Ni-P alloys have good mechanical, electrical, magnetic, and anticorrosive characteristics. The most important aspects of the electroless procedure are the content and properties of the bath solutions. The impact of such circumstances on a variety of substrates, which have a significant impact on the deposition rate, chemical composition structure, and coating film quality, has been investigated extensively in the past [11,12]. Nanoparticles have been used as an additive in electroless Ni-P plating due to the recent advancements in nanoparticle technology and its application across a wide number of industries [13]. To create a composite coating, solid particles, such as alumina, titanium oxide, silicon carbide, and silicon nitride are combined in an electroless coating solution [14]. When compared to planar Ni-P electroless coatings, these coatings exhibit superior physical and mechanical properties. Hardness [15] is one of the most essential attributes that has been found to be important in successful applications. Mechanical properties, like fatigue, corrosion resistance, and creep life are greatly influenced by the microhardness of a surface. Consequently, advances in surface microhardness modeling, and control parameter optimization are required to achieve the optimal level of surface microhardness. It is thus necessary to increase the mean microhardness of the items [16-19]. Therefore, the electroless deposition

including, nano SiC particles on a low carbon steel substrate, and Taguchi approach have been proposed in the present work to enhance the corrosion resistance and mechanical properties of steel.

2. Procedures for Conducting Experiments

2.1 Deposition of the substrate and preparation of the surface

As substrates for this experiment, low carbon steel samples were used, and they performed well. Table 1 provides the chemical composition of the used low carbon steel according to the spectra determined at the Engineering Inspection, and Rehabilitation Company.

Alkaline clean was achieved, with a basic solution (1 M KOH) in order to clean the samples for 15 minutes at the room temperature, and then deionized water was used to finish the rinsing process. In order to prepare the steel samples for the deposition, the following steps were conducted: Using emery sheets, and a 50 % diluted HCl solution, as acid cleaning the whole samples were mechanically polished to a (2000) grade. The samples were first rinsed in water and then dried with a cotton swab. After that, the samples were cleaned, and dried in an oven after being washed with methanol. Right immediately, the samples were immersed in the Ni-P bath. For the nano SiC electroless deposition implanted SiC particles were used in this research, with average nano scale 60 nm. These particles fabricated by Nanjing Nanotechnology Co. Ltd., the bath composition is depicted in Table 2, It is kept at (80 -100) °C, and pH equal to 5 throughout the whole, plating process from

Table 2. Bath composition for Ni-P nano SiC 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 SiC	3.5

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

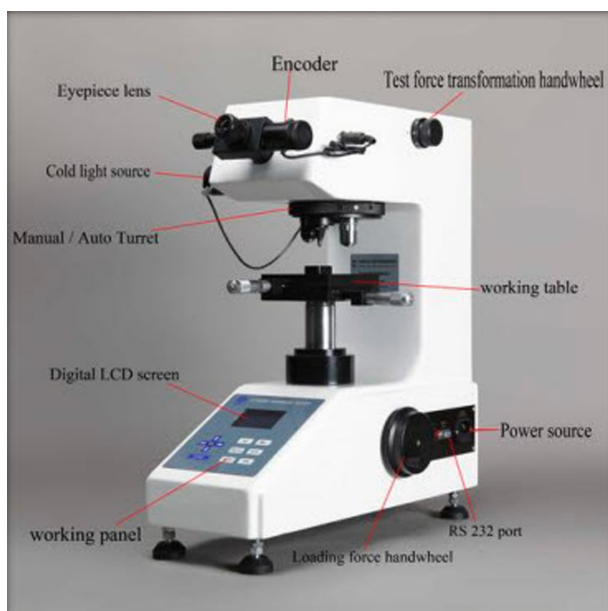


Fig. 1. The microhardness tester

40 minutes to 120 minutes. Each experiment was conducted with a fresh bath solution 200 mL.

2.2 Microhardness

Experiments were carried out on the electroless Ni-P nano SiC coatings. A constant load of 100 g was applied to all deposits to generate indentations, and the hardness values were averaged out of three such determinations. As displayed in Fig. 1, the microhardness test was conducted at the Department of Production Engineering, and Metallurgy - University of Technology - Iraq.

2.3 Weight loss technique

In order to determine the corrosion rate, the mass loss techniques was conducted. It was completed following the recommended technique ASTM [15]. The weight loss techniques have been conducted below distinct temperature situations. The specimens were immersed in a glass cell, in an aerated environment having the HCl without and with the addition of various concentrations of the tested inhibitor to let the solution to be preserved at the required temperature. Before every experiment, the mild steel coupons are regularly polished, completely washed with bi-distilled water, washed with acetone, and dried in the desiccator. The tested coupons were weighed accurately applying an accuracy balance.

Following initial balancing, the coupons were exposed to corrosive media in the absence and presence of the various concentrations of tested inhibitor. After 1, 5, 10, 24, and 48 h of exposure periods, the mild steel coupons were rinsed repeatedly with distilled water, washed with acetone, dried, and weighed. The weight loss analyses have been conducted at temperatures of 30, 40, 50, and 60 °C. All the tests were repeated three times, and the average was taken. The weight loss analyses global principles were based on the determination of the mass loss per specimen regarding the immersion time in HCl media that is maintained at a determined temperature. The corrosion rate was calculated according to equation (1) [19,20]:

$$CR(\text{mm/year}) = \frac{87.600 \times W}{\rho \alpha t}$$

Where C_R is the corrosion rate, W is the average mass loss (g), ρ is the density (g.cm^3), α is the surface area (cm^2), and t is the exposure time (h).

2.4 Taguchi Approach

High quality systems that rely on orthogonal array (OA) trials may be built using the Taguchi method, which is a powerful technique for reducing the variance in testing using the optimal method control settings. It has also been often utilized in engineering studies to improve the performance characteristics by modifying the design factors. In addition to balancing the process parameters and reducing the test runs, orthogonal arrays (OA) are employed. The signal-to-noise (S/N) ratio can be used to evaluate experimental data and extrapolate additional implications. Table 3, quantifies each characteristic's relative relevance [21]. Taguchi approach reduces the number of trials and improves the product quality by utilizing orthogonal arrays, and analysis of variance (ANOVA). The control and noise aspects of the Taguchi concept are often employed to analyze the impact of responses. These considerations are used to choose the best possible process parameters (control parameters), whereas the noise parameters identify any trait that have the potential to cause problems. In addition to the coating factors, and variables, which including bath temperature duration time, coating composition, such as nanomaterials, stabilizer concentration, pH of

the solution, and substrate load, that affected on the characteristics and performance of electroless coatings.

A trial's design and analysis might be quite complicated if all of the factors are taken into account. According to a review of recent publications, researchers most frequently use the three variables, comprising bath temperature (A), plating period (B), and corrosive media (C) to alter the characteristics of electroless nickel deposits [22]. In order to control the rate of reaction, the temperature of the reaction bath must be maintained at an appropriate level, while the speed of stirring was kept

at 500 rpm. As it can be seen in the chart a number of different design characteristics was observed, and they were ranked accordingly. It is possible to analyze the non-linear effects because of the use of three levels.

The degree of freedom (DOF) associated with a given parameter is one less than the number of levels associated with it in the context of design of experiments (DOE). Due to the fact that each of the critical elements is connected to three levels in this scenario, The DOF of each component is two. It is critical to note that the twenty-seven experimental trials stipulated in (OA) are an acceptable number in this circumstance. OA size is necessary for the impact analysis than the total (DOE) size. As a result, for 3-level arrays, the Triangular Table 4 was used to determine L_9 , and its OA factors to the array columns. The OA is the L_9 , OA and the column allocations that go along with it. The values for each cell in the array's three primary parameter columns (A, B, and C) indicate the column's level (1, 2, and 3).

Table 3. 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
Corrosive media C	-	HCl	Na ₂ SO ₄	NaCl

Table 4. L_9 orthogonal array with main parameters

Exp. No.	Corrosive media	Temperature (°C)	Time (min.)
1	HCl	80	40
2	HCl	90	80
3	HCl	100	120
4	Na ₂ SO ₄	80	80
5	Na ₂ SO ₄	90	120
6	Na ₂ SO ₄	100	40
7	NaCl	80	120
8	NaCl	90	40
9	NaCl	100	80

3. Results and Discussion

Table 5 illustrates the values for microhardness, and corrosion rate that were achieved via experiments.

The method is optimized via the use of a desirability strategy. In operations research, desirability is a technique for identifying the operational conditions that have the lowest possible response price. Through the calculation of their desirability (d), where 1 represents the greatest ideal fee and 0 represents the lowest ideal charge [23], each experimental stop result is accurately converted into a scale of [0,1] for a single response. The

Table 5. Experimental microhardness, and corrosion rate values

Exp.No.	Corrosive media	Temperature (°C)	Time (min.)	Speed of stirring	Microhardness (HV)	Corrosion rate (mmpy)
1	HCl	80	40	500	134.267	1.23857
2	HCl	90	80	500	177.200	5.09000
3	HCl	100	120	500	182.911	3.46000
4	Na ₂ SO ₄	80	80	500	147.556	7.12571
5	Na ₂ SO ₄	90	120	500	278.578	2.95429
6	Na ₂ SO ₄	100	40	500	169.667	0.89643
7	NaCl	80	120	500	226.756	4.09714
8	NaCl	90	40	500	109.467	3.93000
9	NaCl	100	80	500	227.467	2.98714

maximum desirability value is then chosen, and the element with the most desirability price is chosen as the most aggregate of characteristics. The reaction is graded into desirability according to the size of answer, with larger being better, smaller being better, and nominal being better. They're explaining it as a large-scale. The-brighter, with larger the better (LTB), the projected reaction's price is likely to be higher than the lower bound. The human desirability feature is described for this response type using the resource of equation (2):

$$d_i(Y)_i = \begin{cases} 0 & Y < L \\ \left(\frac{Y-L}{T-L}\right)^r & L \leq Y \leq T \\ 1 & Y > T \end{cases} \quad (2)$$

It is preferable to be smaller, with nominal the best (STB), and there is a good chance that the estimated response value will fall below the upper limit. The individual desirability functions for this response type are defined in equation (3):

$$d_i(Y)_i = \begin{cases} 0 & Y < T \\ \left(\frac{U-Y}{U-T}\right)^r & T \leq Y \leq U \\ 1 & Y > U \end{cases} \quad (3)$$

For the Better-Nominal-Structure, with smaller the better (NTB), it is expected that the estimated response value will reach a certain target value. The individual desirability for this response type is defined by equation (4):

$$d_i(Y)_i = \begin{cases} 0 & \left(\frac{Y-L}{T-L}\right)^r & Y < L \\ & L \leq Y \leq T \\ \left(\frac{U-Y}{U-T}\right)^r & T \leq Y \leq U \\ 1 & Y > U \end{cases} \quad (4)$$

In these equations, Y represents the responses, U, L, T, and r, r₁, and r₂ represent the weights. Equation (5) is used to determine the composite desirability or the overall desirability after the calculation of individual desirability:

$$D = (d_1 \times d_2 \times d_3 \times \dots \times d_n) = \left(\prod_{i=1}^n d_i \right)^{1/n} \quad (5)$$

Compositional desirability (D) is represented by d₁, d₂, d₃, etc., whereas the maximum desirable values (dn) are represented by the number of answers (n). The experimental values from Taguchi design runs using Minitab

R19 software are analyzed using the complete second degree response surface model supplied by equation (6) [24] :

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta x_i x_i + \sum_{i < j} \beta_{ij} x_i x_j \quad (6)$$

Where, Y represents the answer, x_i represents the ith factor, and k represents the total number of factors. The followings are the corrosion rate and microhardness response surface equations:

3.1 Regression Equation

$$\begin{aligned} \text{Corrosion rate (mpy)} & \quad (7) \\ & = 3.531 - 0.27 \text{ Corrosive media_HCl} \\ & \quad + 0.13 \text{ Corrosive media -Na}_2\text{SO}_4 \\ & \quad + 0.14 \text{ Corrosive media - NaCl} \\ & \quad + 0.62 \text{ Temperature (}^\circ\text{C) - 80} \\ & \quad + 0.46 \text{ Temperature (}^\circ\text{C) - 90} \\ & \quad - 1.08 \text{ Temperature (}^\circ\text{C) - 100 - 1.51 Time (min.)} \\ & \quad - 40 + 1.54 \text{ Time (min.) - 80} \\ & \quad - 0.03 \text{ Time (min.) - 120} \end{aligned}$$

3.2 Regression Equation

$$\begin{aligned} \text{Microhardness} & \quad (8) \\ & = 183.8 - 19.0 \text{ Corrosive media_HCl} \\ & \quad + 14.8 \text{ Corrosive media - Na}_2\text{SO}_4 \\ & \quad + 4.1 \text{ Corrosive media - NaCl - 14.2 Temperature (}^\circ\text{C)} \\ & \quad - 80 + 4.7 \text{ Temperature (}^\circ\text{C) - 90} \\ & \quad + 9.6 \text{ Temperature (}^\circ\text{C) - 100 - 46.0 Time (min.) - 40} \\ & \quad + 0.3 \text{ Time (min.) - 80 + 45.7 Time (min.) - 120} \end{aligned}$$

The preceding equations evince that the electroless plating technique parameters make it difficult to predict the output characteristics based on a simple regression analysis. As a result, the desirability characteristic (D) given by equation (4) is used to determine the most reliable placement of technique parameters to decrease the corrosion rate and maximize the microhardness. The optimal experiments for the corrosive media Na₂SO₄ belonged to experiment 6, that indicated the lowest value for the corrosion rate 0.89643 mpy, while the optimal experiment 5, with highest value for the microhardness 278.578 HV for the same corrosive media. Through the approval function, Fig. 2a, b, and c offers the optimum technology settings for component in order to increase

the microhardness and minimize the corrosion rate. However, the optimal element placement for steel having low carbon with exceptional microhardness (multiple responses) and corrosion charge is given in parenthesis

(c). Table 6 manifests the optimal factor values for each answer, together with their goal value, in order to optimize the desirability function. Because all of the replies are equally valuable, the weight (w_i) value is set to 1.

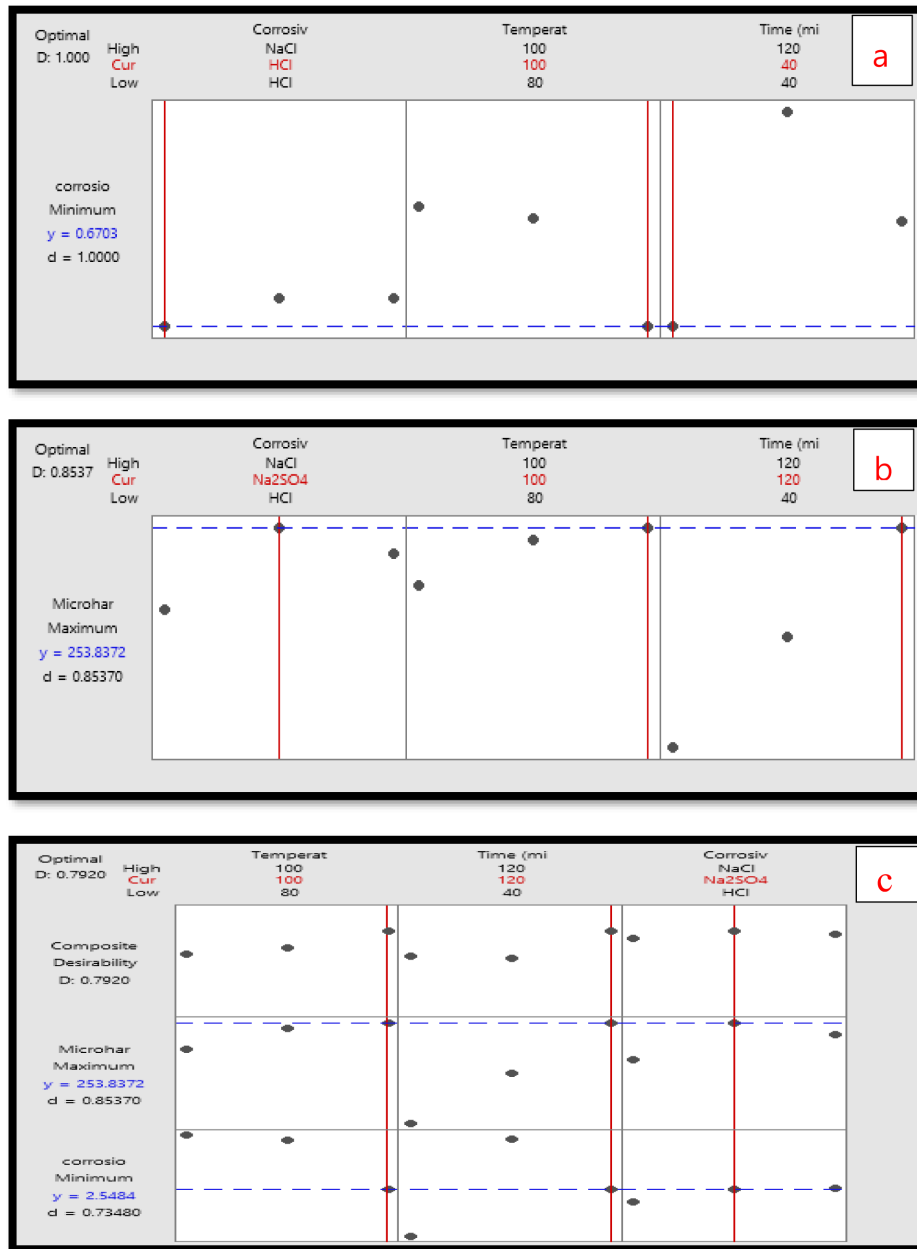


Fig. 2. (a) Maximize the microhardness through desirability function, (b) minimize corrosion rate through desirability function, (c) optimization of multi responses by composite desirability

Table 6. Optimum factors, levels, and target response

Response	Goal	Lower	Target	Weight	Importance
Microhardness	Maximum	109.467	278.578	1	1
Corrosion Rate	Minimum	0.89643	0.89643	1	1

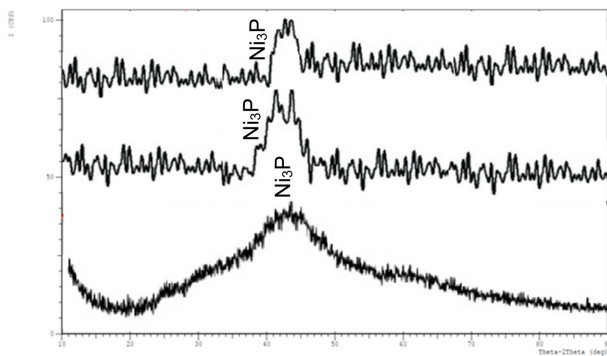
Table 7. Show the optimal factors, and values, as well as the projected response for each variable

Bath temperature (A)	Plating time (B)	Corrosive media (C)	Microhardness	corrosion rate	Composite Desirability
100	120	Na ₂ SO ₄	253.83	2.54841	0.792024

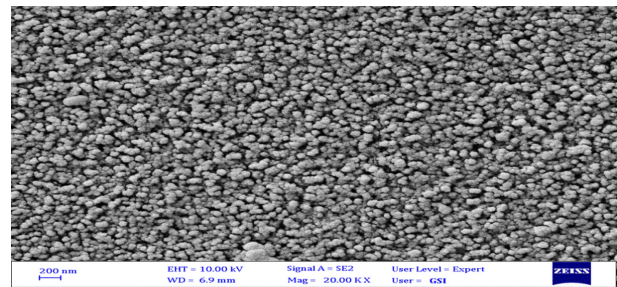
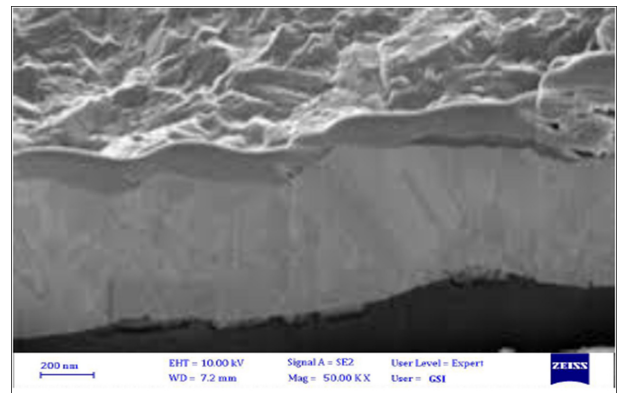
Table 7 elucidates the combined desirability function where one response is maximized and the other is minimized at the same time, as well as the ideal factor values.

3.3 Microstructural investigations

The microstructure of electroless plated steel was examined under optimal circumstances for the material, namely bath temperature 100 °C plating time 120 min, using Ba₂SO₄ as a corrosive medium and Nano-material SiC. The microstructural materials have been identified using X-ray diffraction (XRD) research. The XRD pattern is formed of a collection of amorphous-crystalline peaks under the optimal circumstances (A2B3C2). As seen in Fig. 3, the well-known XRD plot suggests that the Ni₃P coating has a combination of amorphous and crystalline structure. According to the study of the SEM images demonstrated in Fig. 4, the SiC–Ni–P coating applied to the steel Substrate–Ni–P coating interface was free of faults or cracks. Fig. 5 indicated the cross section area of electroless (Ni-P-SiC)

**Fig. 3. XRD pattern of an electroless (Ni-P-SiC) coating**

coating from this figure we can see the uniform and non-porous coating. Statistical reliability was ensured by using the ANOVA method to examine the factorial design study outcomes [22]. The electroless plated samples properties were used to calculate their share of the total electroless-plated low carbon steel reactions to corrosive media were portrayed to be mostly influenced by the ANOVA findings, with an impact ratio of 82%. This is shown in Table 8.

**Fig. 4. Electroless (Ni-P-SiC) coating (SEM)****Fig. 5. The cross section area of electroless (Ni-P-SiC) coating****Table 8. Analysis of variance (ANOVA)**

Source	DF	SS	MS	Contribution%
Corrosive media	2	12590.3	6295.2	82%
Temperature (°C)	2	1791.3	895.7	12%
Time (min.)	2	948.6	474.3	6%
Error	2	6941.3	3470.6	
Total		22271.5		

Where: DF is the, is the degree of freedom, SS is the sum of squares, and MS is the mean square.

4. Conclusions

Using the Taguchi design-based desirability characteristic, the optimization of microhardness, and corrosion rate at a stage in the electroless plating of low carbon steel was achieved. Corrosive media, plating time, and bath temperature were the all system factors that were taken into account. The following findings can be concluded from this investigation:

1. The Ni-P-SiC coating is an aggregation of amorphous and crystalline structures, according to the XRD plot.

2. In order to get the best corrosion resistance and microhardness in the electroless plated steel having low carbon, the following technical characteristics must be met: Na_2SO_4 corrosive medium, plating temperature 100 °C, and plating 120 minutes.

3. In the electroless plated low carbon steel reactions, the corrosive medium was shown to have an effective ratio of (82 %) based only on the findings of the ANOVA analysis.

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