

Improving Wear Resistance of Mechanical Seal Using Ni-P Electroless Coating

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ABSTRACT

Electroless nickel phosphor coating is an important technology in the field of industrial coatings which is widely used in many technical applications. The electroless nickel phosphorus layer has high hardness and corrosion resistance, making it ideal for multiple applications for example, automotive, aerospace and electronics industries. Sulfuric acid is an essential agent in many industrial processes. The corrosive and abrasive nature of sulfuric acid results in rapid wear of mechanical parts, resulting in frequent replacement, increased maintenance costs, and may cause environmental and safety issues. The purpose of this work is to investigate the effect of the different conditions such as heat treatment temperature, coating time and coating bath temperature on wear rate and microhardness by employed electroless Ni-P coating, which was effectively assigned to mechanical seal models. The Taguchi design array L9 was used to conduct the experimental work. The coating thickness was measured using an optical microscope. The results showed that the hardness increased reaching peak as 653 HV at 400 °C and coating time 4 hours. As for wear rate, it was recorded the lowest value as 0.00060 g/m under same conditions. Through SEM and XRD, the formation of NiP and Ni₃P were found. ANOVA demonstrated that heat treatment temperature was the most effected factor that contributed to enhance both hardness and wear resistance, followed by coating time.

Keywords: electroless coating, mechanical seal, wear, sulfuric acid, Ni-P coating.

INTRODUCTION

Sulfuric acid pumps played a major role in the process of transporting high concentration sulfuric acid. However, mechanical seal failure has been encountered, with the maintenance frequency of sulfuric acid pumps increasing per year. Because of the high-temperature mixed acid leaks, this could lower the product's quality and result in accidents [1, 2]. High-frequency maintenance also raises costs and has a significant impact on regular output. As such, it is imperative to elucidate the reasons behind failure and facilitate the quest for answers. There are a lot of coating methods are used to improve the mechanical properties such as, wear resistance, hardness as well as corrosion resistance of components in many industries for example, automotive, aerospace,

petrochemical and so on. One of these forms, electroless coating, is the most significant of the whole bunch [3–5]. Berner and Riedel were used this method for the first time in 1946, they invented it for necessities in many industries. In electroless coating, no electric current was used, only chemical bath, uniform layers of coating are obtained according to the chemical reactions inside the bath [6–8]. Nickel is the most popular type of coating according to specific reasons; it provides a hard surface and high wear resistant. There are three type of Ni coating which are, pure nickel, alloy and electroless coatings. Sodium hypophosphite is responsible for reducing 90% of Ni deposition, which is attributed to its superior corrosion and wear resistance [9]. The electroless Ni-P coating have a lot of good properties such as, bonding strength, weldability, electrical

conductivity and high wear resistance in addition, magnetic properties can control through heat treatment. Heat treatment play a good rule to enhance many properties of the coated specimens [10, 11]. The composition of coated alloy has higher effect on specimen's properties and It can be regulated by modifying the pH, nickel ratio, and bath temperature. The coating thickness, hardness, structure, and morphology of the deposit are significantly influenced by the duration of the coating process and the heat treatment applied [12]. A study by Jensen et al. [13] on the effect of electroless coating thickness on coating corrosion. Ni-Ti particles were added to the coating, then the corrosion rate was measured by measuring the rate of volume loss through wear test, then examining the corrosion paths using an optical microscope. The study found that the coating with Ni-Ti was more uniform in distribution, less cracked, and more resistant to wear. As Li al. [14], they used steel samples Q235 for the coating process, which were $13 \times 13 \times 2$ mm in size. The effect of different annealing temperatures between 200-600 on corrosion resistance was studied and the appropriate temperature was chosen. The results showed that the coefficient of friction decreases when using BN(h) particles with increasing wear resistance, but the hardness decreases with raising the annealing temperature. In general, the corrosion resistance of the coated samples improved at different annealing temperatures as they increased from 2.7×10^{-5} to 7.2×10^{-6} mm³/N m. In this research, Czapczyk et al. [15], the results of the hardness test for electroless plating applied to samples of aluminum used for plastic processing purposes are presented. Three different coating thicknesses were used and their effect regarding the mechanical qualities of the samples, including micro hardness, hardness, and creep, was measured. The Young's modulus of the paint was calculated for each paint thickness. It was found that the hardness increased with the thickness of the paint layer, as it increased from 4000 HM to 6000 HM, meaning it increased by 20%. The effect of heat treatment was examined by Kiran et al. [16] on steel samples AISI 1018 coated with electroless nickel phosphorus and containing boron nitride particles. The findings indicated that an elevation in hardness values was noted due to the influence of the heat treatment temperature. In terms of wear resistance, it was found that corrosion rates decrease at the 400 °C heat treatment temperature as a result of the higher hardness as well as the effect of

lubrication compared to the 300 °C heat treatment, which gave higher wear rates.

Many researchers have investigated the effect of heat treatment on the mechanical properties of the coating, and Ram et al. [17] are one of the researchers who used Ni-P electroless coating on a mild steel specimen. The specimens underwent treatment at 400 °C for one hour. From the results, hardness values increased by 75.5% for the samples that were treated after coating compared to the samples that were not heat treated. In terms of wear resistance, an increase in wear resistance was observed at 400 °C. At a temperature between) 310–330 °C, the nickel-phosphorus phase transforms into Ni₃P [18]. Singh et al [19] estimated the impact of heat treatment and coating time regarding corrosion resistance of iron samples coated with Ni-P iron powder. Investigations indicate that annealing treatment caused in the crystallization of Ni and the precipitation of Ni₃P. The quantity of Ni₃P deposited and the resultant properties change with rising temperature. These changes lead to a clear and noticeable improvement in the microstructure of the coating [20]. Using composite of metallic or nonmetallic materials such as B₄C, CNT and Fe₂O₃ [21-24]. have a positive effect on mechanical properties. Microhardness was improved with high percentage as 240% by 400 °C heat treatment and without using of B4C while, only 127% improvement when 0.3 g/l B4C was used. On the other hand, wear rate was decrease with 90% improvement at 400 °C heat treatment. also, only 84.6% improvement ratio was achieved when 0.7 g/l of B4C was used at same temperature. Contrastively, there was an improvement of wear resistance with 98.8% when CNT composite was used in Ni-B-CNT coating as reinforcement material. Wear resistance when using Fe₂O₃ was higher than Ni-P coating since no appreciable structural change is anticipated at such comparatively low temperatures, The hardness of the coated specimen heat-treated to 200 °C is analogous to that of the as-deposited specimen [25]. However, a significant increase in hardness was observed when the treatment temperature was elevated to around 330 °C (with nearly two hours of annealing), followed by a decline in hardness at higher treatment temperatures. The reduction was attributed to crystal formation, which, after one hour at 400 °C, yielded a peak surface hardness of 916 HK. Buchtík et al. [26] suggested incorporating nickel fluoride or other fluoride

salts into the Ni-P solution to achieve enhanced hardness. It has been determined from the literature that study on Ni-P coating will be done, along with post-processing and sliding wear studies [27]. Ni-P will be electrolessly placed on alloy steel in order to contain the issue. Diffraction analysis is required to support the coat's metallurgical quality, both before to and following sintering. The evaluation of metallurgical bonding and coating adhesion will be conducted through microscopic analysis. Measurements of hardness and wear resistance using the Pin on Disk test are used to assess the resilience of sintered samples to indentation. The wear mechanism and the coating's mechanical qualities as a result of the investigation will be discussed.

EXPERIMENTAL WORK

Material preparation

In this work, the mechanical seal was cut to 9 pieces with dimensions 13×8×8 mm and used in Ni-P electroless coating. The chemical composition was done using device (model) in material college/ metalogical department/ metal Lab. The result is shown in Table 1. The specimens were cleaned firstly and the surface grinding using different grinding papers begging with 180, 220, 400, 600, 800, 1000, 2000 and finely 2500 grid size. Specimens were deposited in Thinner for 30 minutes to clean it from any dust and grinding remnants then rinsed with distilled water through using handed water jet then dried with electrical dryer. Electro cleaning (38% NaOH g/l with 1 L water) was used to clean the specimens from grease and oils with 3V in 70–75 °C for 2 min. Then the specimens rinsed with water and drying. After that, to activate specimens it dipped firstly in 10% HCL for 30 sec to strip it from the oxide layer and secondly the specimens were dipping in activation solution which is consist of (120 ml HCL, 240 g Nickel chloride and the rest is water up to 1 liter) with 5 V DC power supply at room temperature then the specimens were rinsed with water and dried. Activation is necessary step because it increases adhesion between metal and

coating. The chemical compositions of coating bath and its operation condition are illustrated in Table 2. Experiment work of coating is shown in Figure 1. Magnetic stirrer was used with coating bath to keep particles from deposit.

Taguchi design

The Taguchi design array L9 was used to conduct the experimental work in this research with the aim of improving the properties of nickel phosphorus coatings. The Taguchi design allows the analysis of the effect of several variables simultaneously, which minimizes the quantity of experiments needed and increases the efficiency of the research. In this context, three main

Table 2. The composition and process conditions of Ni-P electroless coating

Coating bath composition	
Nickel sulfate	15 g/L
Sodium hypophosphite	30 g/L
Citric acid	20 g/L
Latic acid	20 g/L
Operation conditions	
pH	4–5
Temperature	80, 85, 90 °C
Time	1, 2, 4 hour



Figure 1. Ni-P electroless bath

Table 1. Chemical composition

Element	Fe	Cr	Ni	Mn	Cu	Mo
Wt.%	67.2536	8.7078	10.3406	1.0259	2.7535	9.9132

variables were identified: the temperature during heat treatment, the coating duration, and solution temperature Table 3. By designing various experiments according to the Taguchi matrix, accurate analyses of the interactions between these factors and their effect regarding hardness and wear rate of specimens were achieved. This method contributes to enhancing our understanding of the complex processes related to the properties of materials, allowing the development of more efficient coatings that meet industrial needs, such as mechanical seals for sulfuric acid pumps.

Wear test

To study surface film properties heat treated was done in furnace with Aragon. The samples were thermally heated under different temperature (200, 300 and 400 °C) for 1 hour. Wear rate was measured of base and Ni-P electroless coating specimens against a high speed steel ball have a hardness about 60 HRC with diameter as 6 mm by Pin on Disk method. Rotation speed was 250 rpm and rotational radius was 2 mm. A

10 N load was used. Experiments was done with sliding distance as 62.8 m were made 20 min. the work was done in room temperature. No lubricant was used. The specimen’s weight was measured before and after the test and weight loss was calculated. Wear rate was calculated according to the formula below [28]:

$$Wear\ rate = \frac{wear\ weight\ loss}{sliding\ distance} \tag{1}$$

The Vickers hardness was assessed. using digital display microhardness tester (HVS-1000) at 50 g load and 15 s durations. The results were taken an average of 3 readings. Coating thickness was measured using optical microstructure in material college/metal lab, A 10× magnification was used to achieve good clarity and acceptable accuracy in the measurements.

RESULTS AND DISCUSSION

Following the application of the Taguchi design and the completion of the required mechanical testing, information regarding the characteristics of the nickel phosphorus coatings resulting from various factors was gathered. We were able to comprehend the impact of each heat treatment, coating time, and coating solution temperature on the mechanical performance of the samples by utilizing exact analysis procedures to assess the hardness and wear rate, see Table 4 that clarified the results. From Table 5 we can notice that coating time have the higher effect on coating thickness and have contribution value as 72.73% and P-value 0.007<0.05.

From SEM examination Figure 2, The transition from the glassy to the crystalline phase and crystallization processes take place when the temperature rises. The material becomes harder as a

Table 3. Taguchi design

No.	Heat treatment temp. (°C)	Coating time (hour)	Coating bath temp. (°C)
1	200	1	80
2	200	2	85
3	200	4	90
4	300	1	85
5	300	2	90
6	300	4	80
7	400	1	90
8	400	2	80
9	400	4	85

Table 4. Results for experimental work

No.	Heat treatment Temp. (°C)	Coating time (hour)	Coating bath temp. (°C)	Hardness (g/μm ²)	Wear rate (g/m)	Coating thickness (μm)
1	200	1	80	504	0.00140	13
2	200	2	85	523	0.00134	17
3	200	4	90	548	0.00121	21
4	300	1	85	557	0.00100	14
5	300	2	90	584	0.00088	18
6	300	4	80	601	0.00080	23
7	400	1	90	611	0.00068	18
8	400	2	80	628	0.00069	22
9	400	4	85	653	0.00060	25

result of these changes because they improve the atoms' arrangement in the crystal lattice. Moreover, a compound with a high hardness, Ni₃P may form as the temperature rises as shown in XRD examination Figure 3. The observable increase in

hardness Table 4, is explained by the formation's enhancement of the coating's mechanical strength. Raising the temperature causes the material's internal tensions to decrease, allowing the atoms to rearrange more easily and improving the material's

Table 5. Analysis of variance for thickness

Source	DF	Contribution	Adj SS	P-Value
Heat treatment temp.	2	26.26%	34.6667	0.019
Coating time	2	72.73%	96.0000	0.007
Coating bath temp.	2	0.51%	0.6667	0.500
Error	2	0.51%	0.6667	
Total	8	100.00%		

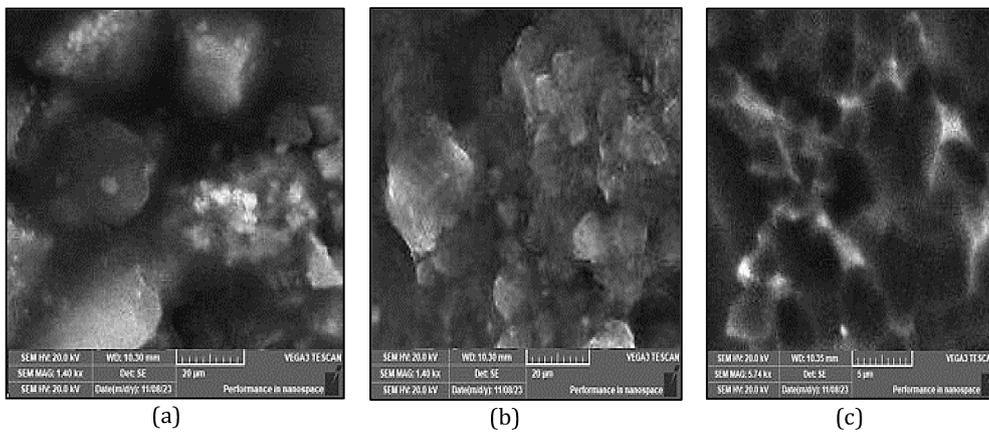


Figure 2. SEM test for specimen after heat treatment: a) 200 °C, b)300 °C and c) 400 °C

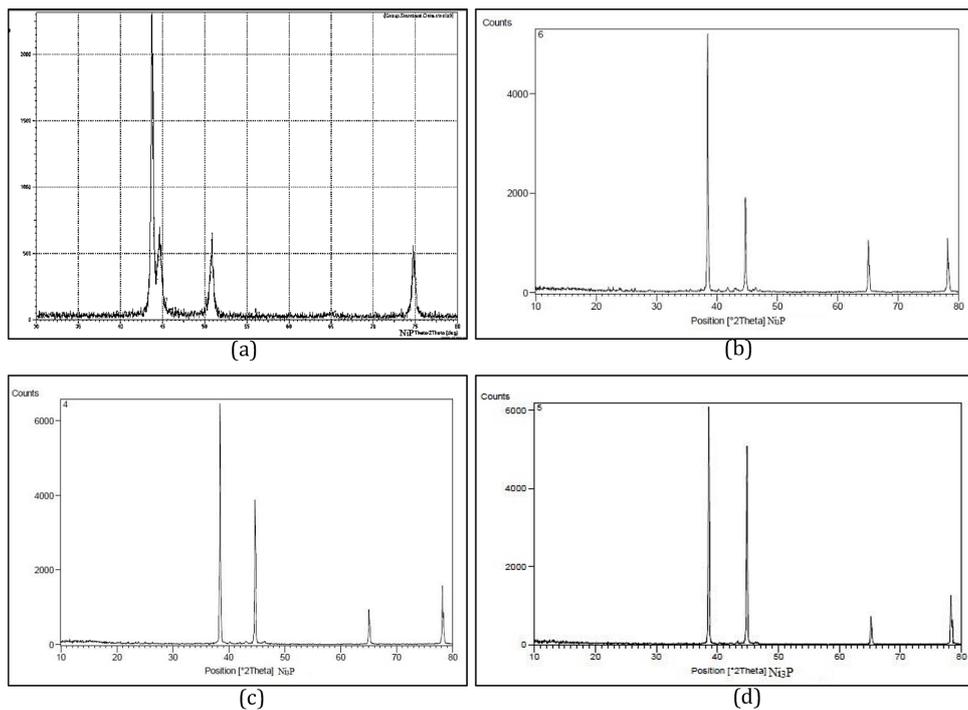


Figure 3. XRD pattern of specimen after heat-treatment: a) as plated, b) 200 °C, c) 300 °C and d) 400 °C

mechanical qualities. It is evident from the analysis of variance, Table 6, that the contribution ratio is 85.46% and the P-Value is 0.001. These numbers show that temperature significantly and favorably affects the coating's hardness. A P-Value below 0.05 signifies that the variation in temperature-dependent hardness is statistically significant. The significance of this variable in enhancing the mechanical properties is confirmed by a high contribution ratio. The coating will stay longer if the coating

period is extended, which will enhance the coating's thickness [19] as shown in Figure 4. Since thicker layers frequently have higher strength and better stress tolerance, increasing the thickness may help to improve the hardness. Better bonding between the nickel and phosphorus atoms is made possible by the extra time, which improves the coating's cohesiveness and hardness, Figure 5 show the mean effect of variables on hardness. P-Value: 0.004 Rate of contribution: 14.36% This finding suggests that

Table 6. Analysis of Variance of microhardness

Source	DF	Contribution	Adj SS	P-Value
Heat treatment temp.	2	85.46%	16764.2	0.001
Coating time	2	14.36%	2817.6	0.004
Coating bath temp.	2	0.11%	22.2	0.342
Error	2	0.06%	11.6	
Total	8	100.00%		

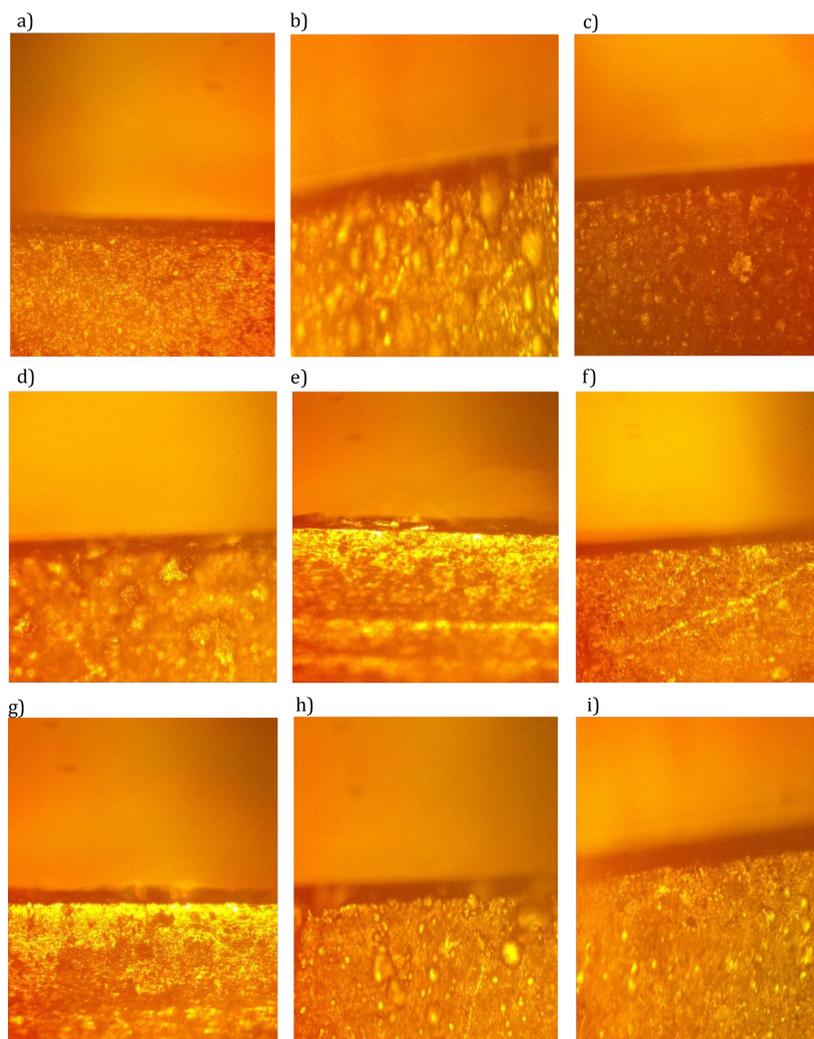


Figure 4. Thickness measurement of specimen according to Taguchi design: a) 1, b) 2, c) 3, d) 4, e) 5, f) 6, g) 7, h) 8, i) 9

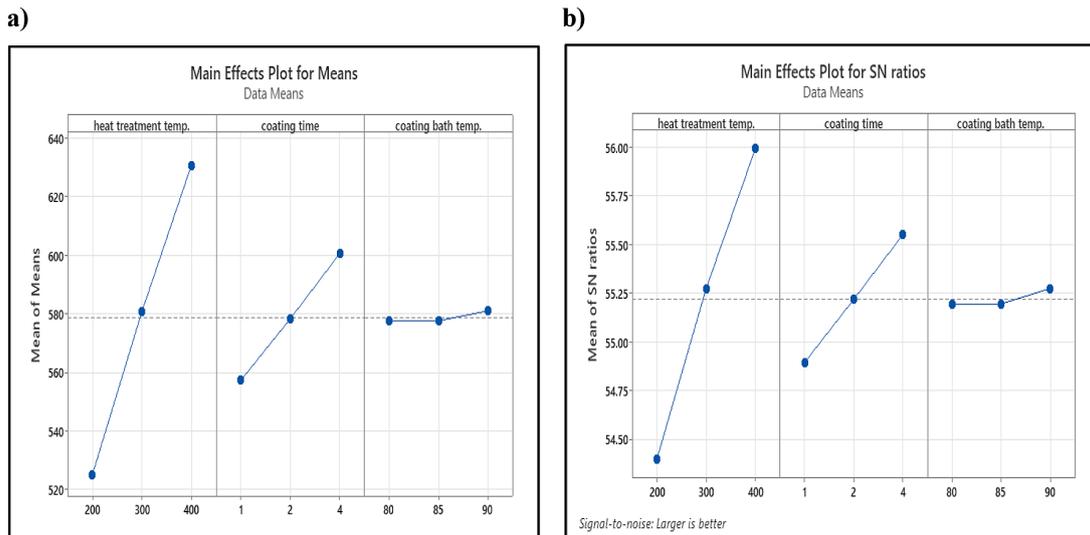


Figure 5. Mean effect plot for hardness: a) means, b) SN ratio

although the influence of coating time is smaller than that of temperature, it still has a considerable impact on hardness. The low P-result result, however, suggests that adjusting the coating time has a favorable impact on the coating’s mechanical qualities. It’s possible that the coating bath’s temperature is within an insufficient range to significantly alter the chemical or physical properties that influence hardness. As a result, the lack of a discernible effect implies that other variables – like the temperature during curing and the duration of the coating are more crucial. P-value is 0.342. Rate of contribution: 0.11% This number suggests that the coating’s hardness is mostly unaffected by the bath’s temperature. A high P-Value indicates that a change in this variable does not result in a statistically significant difference in hardness. Thus, it may be concluded that bath temperature has little bearing on enhancing the coating’s qualities. Heat treatment at elevated temperatures (300 °C and 400 °C) results in a substantial reduction in the wear rate, signifying a notable enhancement in wear resistance. This results from the enhancement of the crystal structure and the augmented creation of interfacial compounds, such as Ni₃P. Heat treatment contributes to

a 93.84% reduction in wear rate, as shown in Table 7, showing its paramount influence on enhancing wear resistance. Heat treatment-induced structural alterations enhance the cohesiveness and strength of the coating. As discussed regarding hardness, an increase in temperature enhances the hardening of the coating, hence mitigating weight loss from corrosion (Figure 6). Research demonstrates that a more uniform crystal structure improves materials’ resistance to friction [2, 6, 22], as evidenced by the wear rate results in Table 1. Extending the coating duration, particularly from 1 hour to 2 hours, results in a notable enhancement in the wear rate, which dramatically declines to 0.00068 after 4 hours of coating. This indicates that prolonging the coating duration facilitates the development of a thicker and denser layer, hence augmenting its corrosion resistance (Figure 6). The percentage contribution to the wear rate is 5.28%, signifying a beneficial influence, albeit less significant than that of the heat treatment. Extending the coating duration allows for prolonged material interaction, resulting in the development of a thicker and denser layer. This layer serves as a protective barrier against corrosion, as thicker layers typically exhibit enhanced

Table 7. Analysis of variance for wear rate

Source	DF	Contribution	Adj SS	P-Value
Heat treatment temp.	2	93.84%	0.000001	0.002
Coating time	2	5.28%	0.000000	0.030
Coating bath temp.	2	0.71%	0.000000	0.185
Error	2	0.16%	0.000000	
Total	8	100.00%		

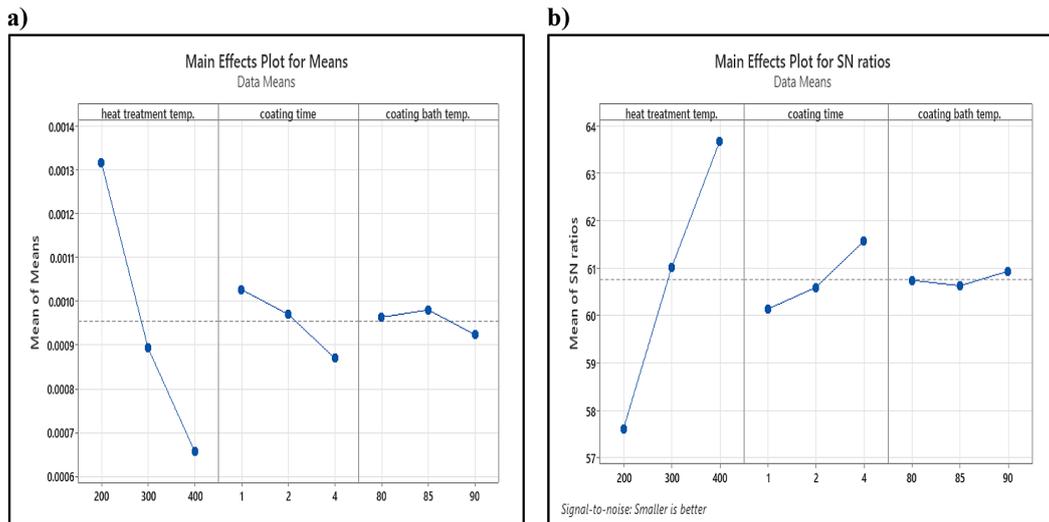


Figure 6. Mean effect of process variables on wear rate: a) means, b) SN ratio.

resistance to wear. The bath temperature shown no impact on the wear rate, contributing merely 0.71%. The temperature range employed may have been inadequate to produce a substantial impact on the coating qualities. The temperature range employed (80–90°C) may be inadequate to induce significant alterations in the coating characteristics. In contrast to prior investigations, which indicate that elevated temperatures may facilitate enhanced outcomes, the present experiment may have been constrained by additional variables, such the concentration of materials in the bath or ambient circumstances.

Looking at the residual plots (Figure 7) of the wear rate, normal Probability Plot, shows the normal

distribution of the residuals of the data. There is a slight deviation at the extreme values, as the data approaches a straight line. As for Versus Fits, since there is no clear pattern, it is evidence that the model is appropriate and there is no bias. Histogram, shows that the values are centered around zero, which is close to the normal distribution. Finally, Versus Order show that there is no temporal correlation with the residuals, due to the lack of a clear pattern. The same results were obtained for hardness as shown in Figure 8. In general, the figures show that the model used to analyze both the wear rate and the hardness is appropriate, which indicates the quality of the results and the analytical model.

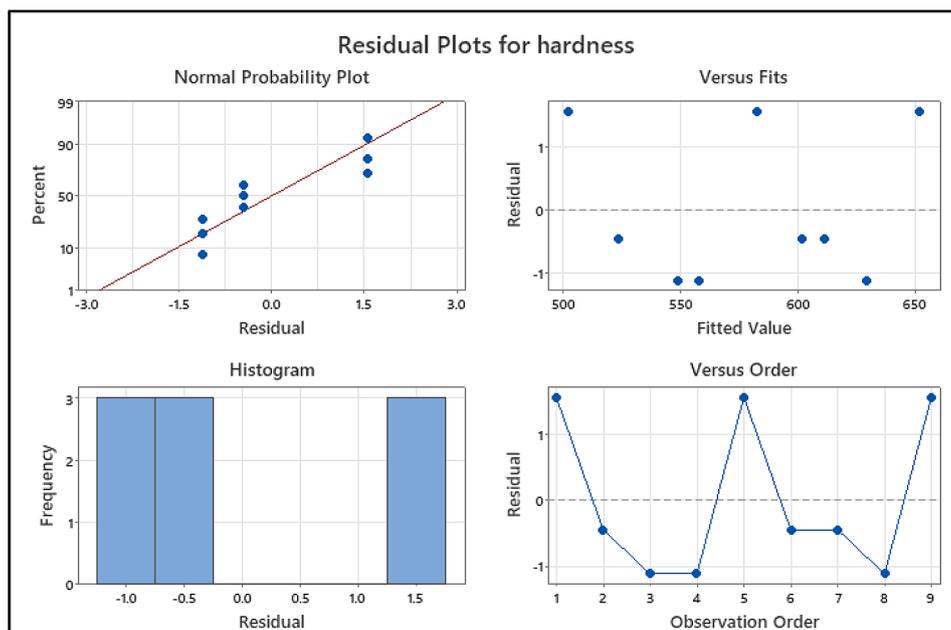


Figure 7. Residual plot for hardness

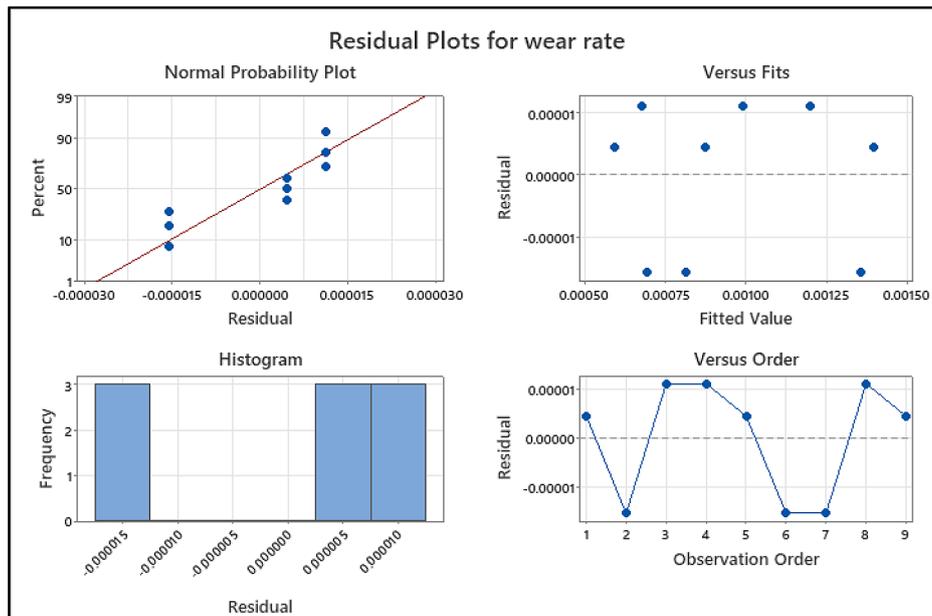


Figure 8. Residual plot for wear rate.

CONCLUSIONS

The research demonstrated the impact of Taguchi design on enhancing the characteristics of nickel phosphorus plating by many factors: heat treatment, plating duration, and plating solution temperature.

1. Hardness: The findings indicated a notable enhancement in hardness with elevated heat treatment temperatures, achieving 653 HV at 400 °C with a plating duration of 4 hours. The results indicated that plating duration positively influenced hardness, which increased with extended time.
2. The wear rate diminished with elevated heat treatment temperatures, with the minimum wear rate measured at 0.00060 g/m at 400 °C and a plating duration of 4 hours. The duration of plating significantly enhanced wear resistance.
3. Analysis of variance: The ANOVA results indicated that the heat treatment temperature exerted the greatest influence on enhancing hardness and wear rate, followed by plating duration, whereas the plating solution temperature had the minimal impact.
4. Structural analysis: Techniques such as XRD and SEM validated the alterations in the coating’s crystal structure, illustrating the correlation between mechanical qualities and structural transformations.

The results indicate that modifying the specified variables significantly enhances the mechanical performance of the coatings, thereby

improving their applicability in industries necessitating high corrosion resistance, such as in mechanical seals for sulfuric acid pumps.

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