

Optimization of Ni–B Coating Process Parameters Through Factorial Design and Taguchi Approach [†]

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Abstract

Protective coatings enhance abrasion and wear resistance, extending component lifespan. This study optimizes bath and heat treatment parameters for Ni–B coatings to improve tribological performance. Synthetic datasets of the coefficient of friction (COF) were analyzed using factorial design and the Taguchi method. The optimal COF values were $\mu_{opt} = 0.4244$ (factorial) and $\mu_{opt} = 0.3998$ (Taguchi), with the latter's confidence interval [0.3671, 0.4325] confirming the validity of both. For minimizing COF, optimal settings were $t_f = 60$ °C, $T_l = 60$ min, $t_h = 200$ °C; for maximizing Vickers microhardness, $t_f = 85$ °C, $T_l = 100$ min, $t_h = 600$ °C, with a confidence interval of [814.17, 867.48]. These findings offer valuable insights into optimizing the Ni–B coating process.

Keywords: factorial design; Taguchi approach; orthogonal array; tribological properties; optimization; analysis of variance (ANOVA); coating; coefficient of friction

1. Introduction

Electroless plating technologies have gained increasing significance in recent years, as coatings produced without electrolysis are being adopted more widely across various industrial sectors. This growing interest underscores the importance of studying the tribological properties of Ni–P and Ni–B coatings, which were investigated in our previous research project [1]. In that work, we presented and characterized the examined samples and reported in detail the results of microhardness measurements, wear tests, and scanning electron microscopy (SEM) analyses. These investigations aimed to determine key tribological parameters of Ni–P and Ni–B coatings, including the coefficient of friction, wear volume loss, and wear rate. The study revealed that the Ni–P coating resulted in the best tribological properties, combining the highest average Vickers microhardness with the lowest measured wear volume and wear rate relative to the frictional work. The results clearly demonstrated that the tribological behavior of electroless nickel-based coatings is significantly influenced by the parameters of the chemical plating bath. This observation highlights the need for further research to determine the optimal bath composition and heat treatment conditions that maximize tribological performance.

Li et al. developed a nickel-based nanocomposite coating using ultrasonic-assisted pulsed electrodeposition [2]. They focused on optimizing deposition parameters to improve hardness and wear resistance, showing the method's effectiveness for surface enhancement. Niksefat and Mahboubi investigated a nickel–boron-based composite coating enhanced



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with reduced graphene oxide under various plasma nitriding durations [3]. Their research demonstrated that surface treatment significantly improved the mechanical and tribological behavior of the nickel-based coating. Reddah et al. optimized the electrodeposition process of nickel–alumina composite coatings using the Taguchi method [4]. Their findings highlighted the influence of process parameters on the structure and hardness of the nickel-based coating, resulting in significant performance gains. Mei et al. provided insights into optimizing a nickel–boron–molybdenum (Ni–B–Mo) electroless coating on GCr15 steel [5]. They showed how salt concentration and deposition time affect the deposition rate and hardness of the nickel-based coating, with the goal of enhancing wear resistance. Chintada et al. provided a comprehensive review of electroless nickel-based coatings, emphasizing the effects of various process parameters on coating properties [6]. They focused on Ni–P and Ni–B composites, underscoring their potential in improving hardness, wear resistance, and corrosion protection. Reddy Paturi et al. provided a review on the use of artificial neural networks for modeling and optimizing surface coating processes, including nickel-based systems [7]. They emphasized the value of machine learning in understanding complex process-property relationships to support the advancement of coating technologies. Together, these sources validate the scientific relevance of applying structured, statistically grounded optimization approaches, even on synthetic datasets, to advance understanding and performance of Ni–B coatings.

The scientific novelty of this work lies in the methodological approach to optimizing the plating bath and heat treatment parameters of Ni–B coatings. While prior studies focused on experimental characterization of electroless coatings, this study applies a full factorial design, Taguchi method, and ANOVA to evaluate the effects and interactions of process parameters on key tribological properties. Furthermore, the use of synthetic datasets for this statistical optimization framework provides a unique methodological contribution that can guide future experimental research by narrowing down the most influential parameter combinations.

To support this research objective, Section 2 reviews the main bath parameters affecting the characteristics of Ni–P and Ni–B coatings and discusses the expected effects of heat treatment based on a review of the relevant literature and the theoretical backgrounds of factorial design, Taguchi method, and analysis of variance (ANOVA). Section 3 applies a full factorial experimental design to identify the optimal plating bath and heat treatment parameters using synthetic datasets. The repeatability of the synthetic datasets is evaluated using the Cochran criterion, while Student's *t*-test determines the significance of regression coefficients, and the adequacy of the response function is assessed using the Fisher criterion. Based on the verified response function, the optimal values for bath temperature, plating time, and heat treatment temperature are determined using Solver Add-in for Excel/Microsoft 365. The optimization procedure is detailed for the coefficient of friction. Section 4 applies the Taguchi method to the experimental design, followed by variance analysis of the obtained results. The ANOVA results indicate that none of the considered factors can be neglected, allowing for the determination of optimal experimental settings for all three factors. Notably, the optimal parameters identified by the Taguchi method are consistent with those obtained via full factorial design, supporting the validity of the results. Section 4 focuses on discussions and future research directions.

2. Materials and Methods

In this Section, we will discuss the impact of bath parameters on the tribological properties of Ni–P and Ni–B coatings, and highlight the main theoretical aspects of factorial design, the Taguchi method, and ANOVA for optimizing bath parameters in Ni–B coating deposition. The data used to build the polynomial model were created artificially, based

on a made-up polynomial function. The reason for using synthetic data was to provide a simple and controlled setting where the use of factorial design and Taguchi methods for parameter optimization could be clearly demonstrated. Since the data were not collected from real experiments, this approach allowed full control over the conditions and outcomes. To make the model more realistic and better reflect the kinds of variations that often occur in real experiments, two different sets of synthetic data were generated. One set represented ideal, error-free values, while the other included synthetic measurement errors. This way, the model could take into account the effect of experimental uncertainty and provide a more practical example of how such methods work in real-life situations.

2.1. Bath Parameters Influencing the Properties of Ni–P and Ni–B Coatings

Several studies in the literature have reported on the properties of boron- and phosphorus-containing electroless coatings. A wide range of factors can influence the deposition of Ni–P and Ni–B coatings, including the composition of the plating solution, pH level, and temperature, all of which significantly affect the physical and mechanical properties of the coatings [8]. The use of stabilizers—such as thiourea [9], lead acetate [10], or maleic acid [11]—can extend the bath's lifetime and influence both the deposition rate and the surface morphology [12]. Surfactants can also enhance deposition conditions, increase the deposition rate, reduce coating porosity, and improve corrosion resistance [13].

Among these parameters, the pH of the plating bath is particularly critical, as it not only influences the resulting microstructure—amorphous coatings tend to form in acidic media, while crystalline structures develop under alkaline conditions—but also affects the hardness of the coating [14].

Czagány et al. [15] provide one of the most comprehensive analyses, which investigates the properties of phosphorus-containing coatings on W302, a widely used hot-working tool steel. This study deposited coatings with phosphorus contents ranging from 6.73 to 22.2 wt%. The results demonstrated that the properties of the coatings vary significantly depending on their phosphorus content. In the case of Ni–B coatings, the measurement parameters are discussed similarly by Henry [16].

According to the literature, the properties of the coatings can also be influenced by post-deposition heat treatment. Heat treatment in the 300–400 °C range can increase hardness by transforming the amorphous Ni–P alloy into crystalline nickel and a hard nickel phosphide phase. As Figure 1 shows, this structural transformation enhances the hardness of the coating, but it also leads to a reduction in corrosion resistance [17,18].

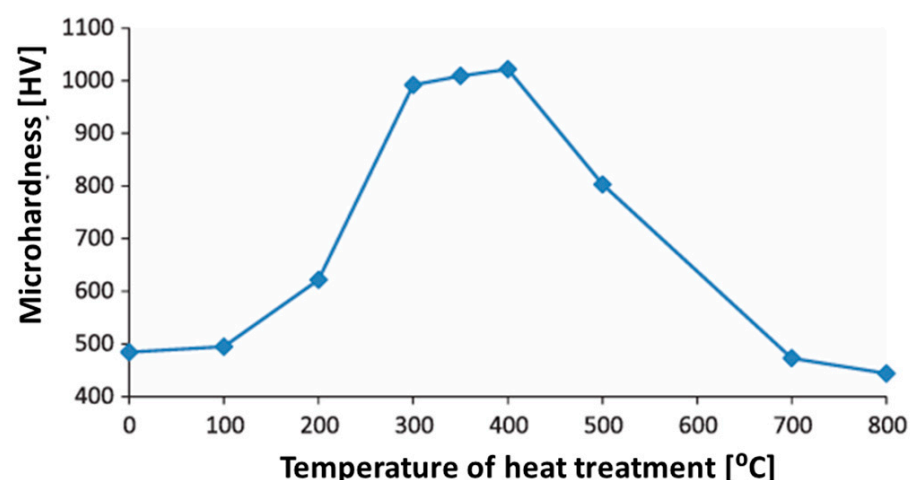


Figure 1. The effect of heat treatment temperature on the hardness of Nickel-Phosphorus coatings.

Based on the above, it can be concluded that there are numerous properties and parameters associated with the plating baths, the deposition process, and the subsequent heat treatment that can influence the tribological behavior of the resulting Ni–B coatings. Among these, it is advisable to focus on those parameters that, according to the literature, significantly impact coating properties and can be adjusted cost-effectively to produce various types of coatings.

Accordingly, Table 1 presents the fixed bath parameters used for coating deposition, while Table 2 summarizes the parameters that can be varied cost-effectively to produce Ni–B coatings with different properties using an alkaline bath.

Table 1. Fixed bath parameters for Ni–B coating deposition in an alkaline bath.

Bath Properties	Ni–B
NiCl ₂ [g/L]	20
C ₂ H ₈ N ₂ [g/L]	90
NaOH [g/L]	90
NaBH ₄ [g/L]	1.2
Thiourea [g/L]	0.001
pH	>12

Table 2. Variable bath and heat treatment parameters for Ni–B coating deposition in an alkaline bath.

Parameter	Recommended Values		
Bath temperature [°C]	60	72.5	85
Deposition time [min]	60	80	100
Heat treatment temperature [°C]	200	400	600

2.2. Factorial Design

Factorial experimental design allows for the simultaneous investigation of interactions between variables, which is particularly advantageous in complex systems where the effects of multiple factors can be studied concurrently. However, a major drawback is that the number of experiments increases exponentially with the number of factors considered, which can quickly become resource- and time-intensive. This limitation can make the study of systems with a large number of factors challenging [19]. The steps of traditional factorial experimental design are defined in a wide range of literature [20,21].

2.3. Taguchi Method

The Taguchi method was developed to improve traditional factorial experimental design, significantly reducing the number of required experiments. Its orthogonal arrays allow complex problems to be studied in a simplified manner. These arrays contain predefined experimental designs that save both time and cost. When discussing the Taguchi method, it is important to clarify two key concepts: a factor and a level. A factor is a variable or element examined in the experiment that can influence the output, while a level refers to the possible values that a given factor can take. For example, water pressure could be a factor in waterjet surface machining, and its levels would be the specific pressure values tested [22]. The experimental design begins with a brainstorming session to identify the most important quality characteristics and the factors to be investigated. Besides brainstorming, one should consider allocating part of the experimental budget to a screening design. Though not suitable for optimization, it helps identify key factors for the Taguchi experiments. Based on the Taguchi “cookbook”, an appropriate experimental plan is then selected and conducted. The optimal levels of the factors are determined during the analysis of the results, which must be validated through a confirmation experiment. Table 3

presents the reference table that facilitates the selection of the appropriate orthogonal array based on the number of parameters and levels, containing the experimental design corresponding to the given parameter and level counts [23].

Table 3. Reference table for selecting orthogonal arrays.

Levels	Number of Parameters (Factors)								
	2	3	4	5	6	7	8	9	10
2	L4	L4	L8	L8	L8	L8	L12	L12	L12
3	L9	L9	L9	L18	L18	L18	L18	L27	L27
4	L16	L16	L16	L16	L32	L32	L32	L32	L32
5	L25	L25	L25	L25	L25	L50	L50	L50	L50

2.4. Analysis of Variance

The method divides the total variance into components and examines whether the variance between groups is significantly greater than the variance within groups. Analysis of variance is particularly useful in research where the effects of multiple independent variables are to be analyzed simultaneously [24].

3. Results

In this Section, we will discuss the application of factorial design, the Taguchi method, and analysis of variance to optimize the bath and heat treatment parameters of Ni–B coatings.

3.1. Application of Factorial Design to Optimize Bath and Heat Treatment Parameters

As the first step of the experimental design, several basic parameters must be defined, which are as follows:

- Number of factors: $f = 3$ (bath temperature, separation time, heat treatment temperature),
- Number of levels for experimental settings: $p = 3$ (for all three parameters, there are three levels according to Table 2),
- Number of independent settings: $n = f^p = 3^3 = 27$,
- Number of repetitions: $m = 2$ (for each test piece, two measurements are performed),
- Number of measurements: $m \times n = 2 \times 27 = 54$.

The data used for the polynomial model were synthetically generated based on a hypothetical polynomial function. The purpose of using synthetic data representing potential outcomes was to demonstrate the application of factorial design and Taguchi methods in parameter optimization within a controlled, idealized framework. Two sets of synthetic data were created to enable the incorporation of measurement error into the model, allowing for a more realistic representation of experimental variability.

The response function depends on the following three parameters: t_f is the bath temperature [°C], T_l is the separation time [min], and t_h is the heat treatment temperature [°C]; therefore, the approximating polynomial for the response function is sought in the following form:

$$\mu(t_f, T_l, t_h) = \beta_0 + \beta_1 \cdot t_f + \beta_2 \cdot T_l + \beta_3 \cdot t_h + \beta_{12} \cdot t_f \cdot t_h + \beta_{13} \cdot t_f \cdot T_l + \beta_{23} \cdot T_l \cdot t_h + \beta_{123} \cdot t_f \cdot T_l \cdot t_h, \quad (1)$$

where β is the coefficient of the response function, μ is the coefficient of friction. Using the full factorial experimental design methodology, we determined the response function in the case of synthetic datasets of coefficient of friction values as follows:

$$\mu(t_f, T_l, t_h) = 2.902 \cdot 10^{-1} + 3.973 \cdot 10^{-4} \cdot t_f + 2.139 \cdot 10^{-4} \cdot T_l + 1.934 \cdot 10^{-5} \cdot t_h + 1.205 \cdot 10^{-5} \cdot t_f \cdot T_l + 2.815 \cdot 10^{-6} \cdot t_f \cdot t_h + 1.38 \cdot 10^{-6} \cdot T_l \cdot t_h. \quad (2)$$

Subsequently, using Excel Solver, we determined the optimal bath and heat treatment parameters. Numerically, we found the following solution: $t_f = 60$ °C, $T_l = 60$ min and $t_h = 200$ °C, which means that the lowest friction coefficient is obtained when the coating is produced in a bath at 60 °C, the separation time is 60 min, and the heat treatment temperature is 200 °C. These optimal parameters correspond to the synthetic datasets.

3.2. Application of the Taguchi Method to Decrease the Number of Experiments

Based on Table 3, for three factors and three levels, the L9(3³) orthogonal array should be selected. The structure of the L9(3³) orthogonal array [25] for analyzing coating process parameters is shown in Table 4.

Table 4. The L9(3³) orthogonal array for the investigation of coating process parameters using synthetic datasets.

Experimental Setup	Factor			Coefficient of Friction [-]
	Bath Temperature [°C]	Deposition Time [min]	Heat Treatment Temperature [°C]	
1	60	60	200	0.4049
2	60	80	400	0.4726
3	60	100	600	0.5390
4	72.5	60	400	0.4655
5	72.5	80	600	0.5429
6	72.5	100	200	0.4623
7	85	60	600	0.5475
8	85	80	200	0.4651
9	85	100	400	0.5511

3.3. Application of Variance Analysis to Optimize Bath and Heat Treatment Parameters

As the first step of the ANOVA method, the sums of the experimental results for each factor at its lower, base, and upper levels are calculated using the following equations:

$$F_{i,1} = \sum_{k=1}^{k_{max,i,1}} y_{i,1,k}, F_{i,2} = \sum_{k=1}^{k_{max,i,2}} y_{i,2,k} \text{ and } F_{i,3} = \sum_{k=1}^{k_{max,i,3}} y_{i,3,k}. \quad (3)$$

where $F_{i,1}$ is the lower level, $F_{i,2}$ is the base level, $F_{i,3}$ is the upper level of factor i , $y_{i,1,k}$ is the result of experiment k conducted at the lower level of factor i , $k_{max,i,1}$ is the number of different experiments performed at the lower level of factor i , $y_{i,2,k}$ is the result of experiment k conducted at the base level of factor i , $k_{max,i,2}$ is the number of different experiments performed at the base level of factor i , $y_{i,3,k}$ is the result of experiment k conducted at the upper level of factor i , and $k_{max,i,3}$ is the number of different experiments performed at the upper level of factor i .

Then, the average results for each factor at these levels are determined as follows:

$$\bar{F}_{i,1} = \frac{F_{i,1}}{k_{max,i,1}}, \bar{F}_{i,2} = \frac{F_{i,2}}{k_{max,i,2}} \text{ and } \bar{F}_{i,3} = \frac{F_{i,3}}{k_{max,i,3}}. \quad (4)$$

where $\bar{F}_{i,1}$ is the average of measured values on the lower level, $\bar{F}_{i,2}$ is the average of measured values on the base level, and $\bar{F}_{i,3}$ is the average of measured values on upper levels. Next, the effect of each factor on the experimental results is evaluated based on the absolute difference $|\bar{F}_{i,1} - \bar{F}_{i,3}|$. As shown in Table 5, the heat treatment temperature has the greatest influence on the measured coefficient of friction, since $|\bar{F}_{3,1} - \bar{F}_{3,3}| > |\bar{F}_{2,1} - \bar{F}_{2,3}| > |\bar{F}_{1,1} - \bar{F}_{1,3}|$.

Table 5. Analysis of the effect of each factor on the experimental results.

Statistical Characteristic		Factors		
Name	Symbol	t_f [°C]	T_l [min]	t_h [°C]
Sum of the results of experiments (lower levels of factors)	$F_{i,1}$	1.4165	1.4178	1.3324
Sum of the results of experiments (base levels of factors)	$F_{i,2}$	1.4707	1.4807	1.4892
Sum of the results of experiments (upper levels of factors)	$F_{i,3}$	1.5637	1.5524	1.6294
Average of the results of experiments (lower levels of factors)	$\bar{F}_{i,1}$	0.4722	0.4726	0.4441
Average of the results of experiments (base levels of factors)	$\bar{F}_{i,2}$	0.4902	0.4936	0.4964
Average of the results of experiments (upper levels of factors)	$\bar{F}_{i,3}$	0.5212	0.5175	0.5431
Difference between the averages of the lower and upper level experiments	$ \bar{F}_{i,1} - \bar{F}_{i,3} $	0.0491	0.0449	0.0990

Subsequently, the sum of squares for each factor can be determined, followed by calculating the sum of squares for the error term according to the equation (see Table 6). The degrees of freedom for each factor are also determined, and the degrees of freedom for the error are calculated. With this information, the variance values for the factors can be calculated, while the variance of the error is also computed.

Table 6. Sum of squares, degrees of freedom, and variance values for factors and the error.

Statistical Characteristic		Factors			Error
Name	Symbol	t_f [°C]	T_l [min]	t_h [°C]	
The sum of squares for each factor and the error	S_i, S_e	0.0037	0.0030	0.0147	0.00014
Degrees of freedom for each factor and the error	f_i, f_e	2	2	2	2
Variance values for each factor and the error	V_i, V_e	0.0018	0.0015	0.0074	0.00007

Subsequently, the variance ratios of the factors can be determined based on the variance values of the individual factors and the error term. The pure sum of squares for the factors is calculated, and the percentage contributions of the factors are computed. The calculation results are summarized in Table 7.

Table 7. Calculated values of variance ratios, pure sums of squares of factors, and percentage contributions of factors.

Statistical Characteristic		Factors		
Name	Symbol	t_f [°C]	T_l [min]	t_h [°C]
Variance ratio of the individual factors	VA_i	25.55	20.90	101.75
The pure sum of squares of the individual factors	S'_i	0.0036	0.0029	0.0146
Percentage contribution of the individual factors	P_i	16.46%	13.34%	67.53%

Table 7 shows that the heat treatment temperature influenced the friction coefficient value by 67.53%, the bath temperature by 16.46%, and the deposition time by 13.3%. Since none of the factors' effects are negligible, and considering that the "smaller is better" principle applies to the friction coefficient, the optimal experimental settings are as follows: bath temperature $t_f = 60$ °C, deposition time $T_l = 60$ min and heat treatment temperature $t_h = 200$ °C.

The expected value of the experiment result for the optimally selected experimental settings was determined: $y_{opt} = 0.3998$, and the confidence interval of the expected value was calculated: $KI = \pm 0.0327$.

The estimated optimal value ($y_{opt} = 0.3998$) is very close to the best measured value ($y_{min} = 0.4049$), indicating that, according to the model, the optimal point is not exactly at the measured point but somewhere near or between them. The confidence interval for the optimal value is $[0.3671, 0.4325]$, which includes the best measured value $y_{min} = 0.4049$.

There is no significant difference between the model-estimated optimal value and the best measured value because the best measured value lies within the confidence interval. Statistically, this means that the direction of optimization suggested by the model was correct, but we cannot say with 100% certainty that a better value than the measured one exists.

4. Discussion and Conclusions

It can be stated that the optimal experimental settings determined by the full factorial design coincide with the optimal settings calculated by ANOVA following the Taguchi method. In the optimization based on the full factorial design, the optimal friction coefficient value is $\mu_{opt} = 0.4244$, while according to the ANOVA, following the Taguchi method, the value is $\mu_{opt} = 0.3998$. However, it is important to note that the confidence interval $[0.3671; 0.4325]$ applies only to the $\mu_{opt} = 0.3998$ value. Furthermore, it can be concluded that the optimal friction coefficient calculated through the full factorial design optimization falls within the confidence interval calculated by ANOVA after the Taguchi method, thus confirming the validity of that value. For minimizing the COF, the optimal settings were $t_f = 60$ °C, $T_l = 60$ min, and $t_h = 200$ °C. Applying the above method to maximize Vickers microhardness, the optimal experimental settings were $t_f = 85$ °C, $T_l = 100$ min, and $t_h = 600$ °C, which matches the result obtained through the Taguchi method. It is worth noting that the lower bound of the confidence interval $[814.17; 867.48]$ is higher than the best synthetic data (the highest value of Vickers microhardness), indicating that it can produce a coating harder than the synthetic datasets show.

In the future, we would like to test the application of full factorial design and the Taguchi method with simulated data and data based on real measurements. In our opinion, the techniques developed in this thesis can significantly contribute to the rapid evaluation of real experimental results.

It is essential to highlight that the analysis was based on synthetic datasets representing potential outcomes. As such, the findings are intended solely to demonstrate the applicability of statistical optimization methods (e.g., factorial design and Taguchi approach) and do not reflect the real tribological behavior of Ni–B coatings; experimental validation is strongly recommended for future studies.

A potential direction for further research could be the investigation of the effects of different lubricants and the examination of how additional bath parameters influence tribological properties.

In the field of parameter optimization, one possible next step is multi-objective optimization, as in this thesis, the optimization of bath and heat treatment parameters was always based on a single objective function.

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