

Article

Experimental Investigation and Optimization of the Electrodeposition Parameters of Ni-Al₂O₃ Composite Coating Using the Taguchi Method

Ilias Reddah ^{1,2,*}, Laala Ghelani ^{1,2}, Sofiane Touati ¹, Farid Lekmine ¹, Pavol Hvizdoš ³ , Susana Devesa ^{4,*}  and Haithem Boumediri ⁵ 

- ¹ Mechanical Engineering Department, Abbes Laghrour University Khenchela, P.O. Box 1252, Khenchela 40004, Algeria; ghilani_laala@univ-khenchela.dz (L.G.); sofiane.touati@univ-khenchela.dz (S.T.); farid.lekmine@univ-khenchela.dz (F.L.)
 - ² Laboratory of Engineering and Sciences of Advanced Materials (ISMA), Abbes Laghrour University Khenchela, P.O. Box 1252, Khenchela 40004, Algeria
 - ³ Institute of Materials Research, Slovak Academy of Sciences, Watsonova 47, 040 01 Košice, Slovakia; phvizdos@saske.sk
 - ⁴ CEMMPRE, ARISE, Department of Mechanical Engineering, University of Coimbra, Rua Luís Reis Santos, 3030-788 Coimbra, Portugal
 - ⁵ Mechanical Department, Institute of Applied Science and Technology, University of Constantine 1, Constantine 25017, Algeria; haithem.boumediri@umc.edu.dz
- * Correspondence: ilias.reddah@univ-khenchela.dz (I.R.); sdevesa@dem.uc.pt (S.D.)

Abstract: In this work, an experimental investigation is conducted with the aim of optimizing the electrodeposition parameters for Ni-Al₂O₃ composite coatings using the Taguchi method. The presented research is structured into two complementary sections. The first segment investigates the characteristics of Ni and Ni-Al₂O₃ coatings, specifically Al₂O₃ particle incorporation and crystallinity variations, using X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), and hardness evaluation through micro-indentation testing. The second section uses statistical techniques, specifically Analysis of Variance (ANOVA) and signal-to-noise (S/N) ratio analysis, to determine which parameters have the most impact on the experimental results. ANOVA and the Response Surface Methodology (RSM) were used in a modeling technique to forecast and optimize the technical responses. Based on an L16 orthogonal design, sixteen tests were carried out to investigate the effects of several important variables, including agitation rate (200–350 rpm), deposition period (15–60 min), alumina concentration (10–25 g.L^{−1}), and current density (2–5 A.dm^{−2}). The conditions for optimizing microhardness (HV) and Al₂O₃ integration while limiting average crystallite size (ACS) were identified using the most suitable function. The obtained results reveal significant improvements in the composite coating's properties, including a 164% increase in microhardness, a 400% rise in alumina incorporation, and a notable reduction in crystallite size, demonstrating the efficacy of the electrodeposition process and optimization strategy adopted.

Keywords: electrodeposition; Ni-Al₂O₃ coatings; optimization; microhardness; Taguchi method; response surface methodology



Academic Editors: Flavio Deflorian and Uta Klement

Received: 24 February 2025

Revised: 15 April 2025

Accepted: 16 April 2025

Published: 18 April 2025

Citation: Reddah, I.; Ghelani, L.; Touati, S.; Lekmine, F.; Hvizdoš, P.; Devesa, S.; Boumediri, H. Experimental Investigation and Optimization of the Electrodeposition Parameters of Ni-Al₂O₃ Composite Coating Using the Taguchi Method. *Coatings* **2025**, *15*, 482. <https://doi.org/10.3390/coatings15040482>

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

In contemporary industries, various techniques are implemented to protect materials from degradation imposed by chemical and mechanical damage as the wear and corrosion of metal components lead to substantial financial losses globally [1]. To address these

issues, researchers have focused on surface functionalization technologies through surface treatments such as the deposition of thick, thin, or multilayer coatings. An important key development is the application of functionally graded materials (FGMs), which exhibit different properties through their thickness, making them attractive for a range of engineering applications. Thick FGMs are typically produced using techniques such as powder metallurgy, additive manufacturing, and centrifugal casting, while thin FGMs are fabricated through processes including spraying, cladding, chemical vapor deposition (CVD), physical vapor deposition (PVD), atomic layer deposition (ALD), and electrochemical deposition [2–4].

Among the electrodeposition coatings, electrodeposited Ni-Al₂O₃ composite stands out as one of the most widely used nickel-based coatings, which are known for their excellent wear and corrosion resistances, high hardness, and favorable electrical and magnetic properties [5,6].

Recent research has focused extensively on nanocomposite coatings, with particular attention to the fact that the electrodeposition of nickel is significantly influenced by deposition variables and bath chemistry, which determine the nucleation and growth mechanisms. These factors critically impact the texture orientation of the resulting coatings, thereby affecting the microstructure and overall performance of the deposits [7–9]. For example, Wu et al. [10] investigated the impact of boric acid (H₃BO₃) concentration on the texture and microstructural evolution of nickel electrodeposits. This study revealed that varying the H₃BO₃ content in a Watts-type bath could control the preferential orientation of the nickel crystals, significantly affecting the coatings' hardness, residual stress, and abrasion resistance. They found that a higher concentration of H₃BO₃ resulted in a transition from (200) to (220) orientation, with corresponding changes in the mechanical properties.

When it comes to wear resistance, Raghavendra et al. [11] focused on the wear behavior of Al 6061 electrodeposited with Ni- α -Al₂O₃ nanocomposite. They stated that temperature had the most significant effect on the specific wear rate, with the formation of a wear-protective glaze layer up to a critical temperature of 80 °C. This finding underscores the critical role of temperature in wear mechanisms, particularly in high-temperature applications.

Also, the integration of pulse electrodeposition techniques has further pushed the boundaries of nickel coating technology. Karthik et al. [12] applied pulse electrodeposition for composite coatings, emphasizing the critical role of current density, duty cycle, and frequency in grain refinement. Their findings indicate that these parameters significantly influence the tribological properties and wear resistance of the coatings, making pulse electrodeposition a valuable method for producing high-performance materials at a reduced cost.

To fully harness the potential of deposition techniques, advancements in engineering have led to the development of methodologies for material optimization. Taguchi's method, a robust statistical optimization tool, can be effectively applied to identify and classify the critical factors influencing experimental responses based on their significance [13–18].

S.T. Aruna et al. [5] studied the electrodeposition of Ni composite coatings containing yttria-stabilized zirconia (YSZ) particles. Through a Taguchi design and ANOVA, they highlighted the significant roles of particle concentration, current density, and deposition time in determining the microhardness and thickness of the coatings. Their predictive models showed high accuracy in correlating these parameters, emphasizing the importance of deposition conditions in tailoring the characteristics of coatings.

Further advancements in optimization techniques were demonstrated by Yassine Abdesselam et al. [6], who proposed a multi-input–output system based on multiple linear regressions and a genetic algorithm for optimizing the electrodeposition parameters of Ni-P-

Y₂O₃ composite coatings. Their experimental results showed a strong correlation between the predicted and real values, with the model yielding a highly efficient electrodeposition process and superior microhardness compared to similar coatings.

This approach was further validated by Adda Doumi et al. [19], who investigated Ni-P-Al₂O₃ composite coatings, revealing that heat treatment, performed at an annealing temperature of 500 °C, significantly improves the microhardness and corrosion resistance by densifying the coating structure, which was initially porous due to particle distribution issues. The response surface method (RSM) used in this study demonstrated excellent predictive capabilities for optimizing the coating properties.

This study aims to model and optimize the performance of Ni-Al₂O₃ composite coatings deposited by electrodeposition using the partial Taguchi method. This experimental approach offers a dual advantage: identifying the most influential process parameters while reducing the number of required experiments. By simultaneously optimizing multiple factors, the Taguchi method helps minimize result variability, enhance process robustness, and ensure greater reproducibility. Unlike conventional experimental approaches, which typically analyze one factor at a time, this method provides a comprehensive evaluation of parameter interactions, leading to a more efficient optimization of the electrodeposition process.

Initially, the study examines the deposition of pure nickel coatings, analyzing their microstructural behavior and crystallographic orientations. It then extends to Ni-Al₂O₃ composite coatings under different electrodeposition conditions. The optimization process relies on the partial Taguchi method (L16), incorporating four key factors—electrodeposition time, current density, Al₂O₃ concentration, and agitation rate—to assess their influence on microhardness, the weight percentage of incorporated alumina particles, and crystallite size.

The novelty of this work lies in applying the Taguchi method to fabricate Ni-Al₂O₃ composite coatings in a chloride-based electrochemical bath, using predefined process parameters and evaluating the resulting properties through statistical approaches.

The application of the designed experiment and the integration of statistical analysis in this deposition technique represent a significant advancement over traditional experimental methods, providing predictive models and optimized conditions that enable better control and results in high-quality and efficient deposited coatings. Additionally, the models developed in this study have the ability to be transferred to the industry, optimizing manufacturing processes and enabling cost reductions.

2. Materials and Methods

2.1. Materials

Medium carbon steel discs were prepared for electrodeposition, with the detailed substrate preparation, cleaning, and electrodeposition processes outlined below. The coating synthesis was carried out using the traditional Watts bath (Table 1) as the electrolyte, with the substrates connected to the cathode (negative pole) and a high-purity nickel plate (99.8%) serving as the soluble anode (positive pole). Aluminium oxide (Al₂O₃) powder was used as an additive. All chemicals were purchased from Biochem Chemopharma (Quebec, QC, Canada). The process was conducted under the application of direct current (DC), ensuring controlled deposition conditions. The distance between the electrodes during electrodeposition was maintained at 2 cm, with the electrodes fixed using plexiglass supports.

Table 1. Constituents of the traditional Watts bath solution.

Chemical Reagents	Concentration (g.L ⁻¹)
NiCl ₂ ·6H ₂ O	23.77
NH ₄ Cl	21.4
H ₃ BO ₃	18.54

2.2. Substrate Preparation and Electrodeposition Parameters

The steel substrates were cut into discs with a diameter of 20 mm and a thickness of 4 mm using a STRUERS ACCUTOM-100 laboratory chainsaw to ensure identical dimensions. After cutting, the discs were embedded in phenolic resin using an automated hot mounting press to define a working surface area of 3.14 cm². To provide a clean and consistent surface for electrodeposition, the substrates were polished to a mirror finish after embedding.

The polished substrates were then cleaned and degreased by immersing them in acetone for one minute, and then rinsing them with distilled water at room temperature for another minute. Next, to eliminate surface oxides and impurities, the substrates were submerged in a 50 g/L hydrochloric acid pickling solution at 40 °C for one minute. This was followed by a one-minute rinse in distilled water, after which the substrates were dried before proceeding to the next step.

To establish an electrical connection, the resin-embedded substrates were pierced laterally, and copper electrical contact wires were soldered to ensure good conductivity during electrodeposition. The specimens were processed using the co-electrodeposition process with the following factors: current density (A), alumina concentration (B), deposition time (C), and agitation rate (D). Each factor had four levels, as summarized in Table 2.

Table 2. Factors and levels used for electrodeposition of Ni-Al₂O₃ composite coatings.

Factor	Representation	Level 1	Level 2	Level 3	Level 4
Current density	A	2	3	4	5
Alumina concentration	B	10	15	20	25
Deposition time	C	15	30	45	60
Agitation rate	D	200	250	300	350

A 200 mL Pyrex glass beaker was used as the container for the electrolyte solution, which was continuously stirred by a magnetic stirrer bar (6 mm diameter × 30 mm length) at a speed of 600 rpm for 16 h to ensure homogeneity. Before electrodeposition, the solution was subjected to ultrasonic agitation for one hour to eliminate any remaining undissolved particles. The electrodeposition process was conducted using a hot plate stirrer (D_LAB, MS-Pro+) coupled with a thermometer to maintain the required temperature and agitation rate.

The co-deposition of nickel and aluminum oxide particles was performed using the previously prepared steel substrates. After deposition, to remove any loosely adherent alumina particles from the coating, the samples were immediately submerged in deionized water and then ultrasonically cleaned for one minute.

The experimental setup used for the co-electrodeposition process is shown in Figure 1.



Figure 1. Experimental setup used in this study.

2.3. Methods

The Taguchi design is a specialized experimental methodology widely used in engineering and various other fields to refine, through optimization and simplification, different experimental processes, ensuring effective testing with fewer trials. This robust design approach reduces the number of tests while still providing a thorough assessment of crucial process parameters.

The experimental layout is shown in Table 3.

Table 3. L16 experimental layout.

Experiment	Current Density (A.dm ⁻²)	Column Numbers, Deposition Parameters, and Factor Allocation						
		Coded Values	Alumina Concentration (g.L ⁻¹)	Coded Values	Deposition Time (min)	Coded Values	Agitation Rate (rpm)	Coded Values
1	2	1	10	1	15	1	200	1
2	2	1	15	2	30	2	250	2
3	2	1	20	3	45	3	300	3
4	2	1	25	4	60	4	350	4
5	3	2	10	1	30	2	300	3
6	3	2	15	2	15	1	350	4
7	3	2	20	3	60	4	200	1
8	3	2	25	4	45	3	250	2
9	4	3	10	1	45	3	350	4
10	4	3	15	2	60	4	300	3
11	4	3	20	3	15	1	250	2
12	4	3	25	4	30	2	200	1
13	5	4	10	1	60	4	250	2
14	5	4	15	2	45	3	200	1
15	5	4	20	3	30	2	350	4
16	5	4	25	4	15	1	300	3

Key factors influencing the coating properties, such as microhardness, crystallite size, and alumina content, were analyzed through signal-to-noise (S/N) ratios as a logarithmic transformation using the following equations [20]:

$$S/N_{\text{Larger}} = -10 \log 10 \left[\frac{1}{n} \sum_{i=1}^n \frac{1}{Y_i^2} \right] \quad (1)$$

$$S/N_{\text{Larger}} = -10 \log 10 \left[\frac{1}{n} \sum_{i=1}^n Y_i^2 \right] \quad (2)$$

Here, S/N represents the signal-to-noise ratio (in decibels), n denotes the number of repetitions for each experiment, and Y refers to the response values measured during the tests.

In this study, the “larger is better” criterion was applied to maximize microhardness and alumina content, whereas the “smaller is better” criterion was utilized to minimize crystallite size. This systematic approach enabled the identification of optimal parameter combinations while reducing variability caused by external disturbances. The Taguchi design proved instrumental in enhancing the quality and performance of the composite coatings while minimizing experimental resources.

ANOVA analyses were conducted to identify the most influential factors [21] affecting the responses: microhardness, alumina content, and crystallite size. The F-value and p -value derived from the ANOVA were used to assess the significance of each parameter for these responses. Mathematical modeling further enabled the identification of similarities between predicted and experimental outcomes. Additionally, the optimal parameter levels were determined using the desirability function, which aimed to simultaneously maximize microhardness and alumina content while minimizing crystallite size (ACS).

2.3.1. Morphology and SEM Examinations

A scanning electron microscope, SEM, (Thermo Fisher Apreo 2 C, Waltham, MA, USA, equipped with an FEG–Schottky-type electron source, operated at 10.00 kV) and an energy-dispersive X-ray spectroscopy, EDS, system (Oxford™) were employed to analyze the surface morphology and chemical composition of the coatings, respectively. The weight percentage of Al₂O₃ particles incorporated into the Ni matrix was determined. To ensure accuracy, the average weight percentage of Al₂O₃ particles in the Ni/Al₂O₃ coatings was obtained based on SEM microstructure analysis of three selected areas. The coating morphology was further examined using a high-resolution SEM (FEI QUANTA 3D FEG).

2.3.2. XRD Analysis

The X-ray diffraction analysis was carried out using a PANalytical X'Pert PRO diffractometer with Cu K α radiation ($\lambda = 0.15406$ nm). The operating conditions were set at 40 kV and 40 mA, covering a 2θ range from 10° to 80° with a scanning speed of 5°/min. Data collection employed a step size of 0.02° and an exposure time of 1 s per step. The crystallite size of the matrix was calculated using the Debye–Scherrer equation, based on the full width at half maximum (FWHM) of the dominant peak:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (3)$$

where λ denotes the wavelength of the X-ray beam (0.15406 nm), θ is the diffraction angle, K is a constant that depends on the geometry of the crystallite (in this case, 0.89), and β represents the FWHM (in radians).

2.3.3. Hardness Tests

The Vickers hardness of the coated substrates was measured using an LECO LM700AT automatic microhardness tester. A diamond pyramid indenter with an angle of 136° was employed, applying a load of 50 g for an indentation time of 15 s. To ensure accuracy, the microhardness values were calculated as the mean of 10 measurements taken at different locations across the coating surface. This procedure follows the guidelines outlined in ASTM B 57 for the hardness testing of electrodeposited coatings.

3. Results

3.1. XRD Analysis

Figure 2 presents the XRD patterns of pure nickel. The diffraction peaks at 2θ positions corresponding to 44.64° , 52.04° , and 76.51° are indexed to the (111), (200), and (220) planes, respectively, indicating a face-centered cubic (FCC) crystalline structure. The intense peak at (111) suggests a preferential growth orientation. This single phase with an FCC structure was consistent with the results of [22–25].

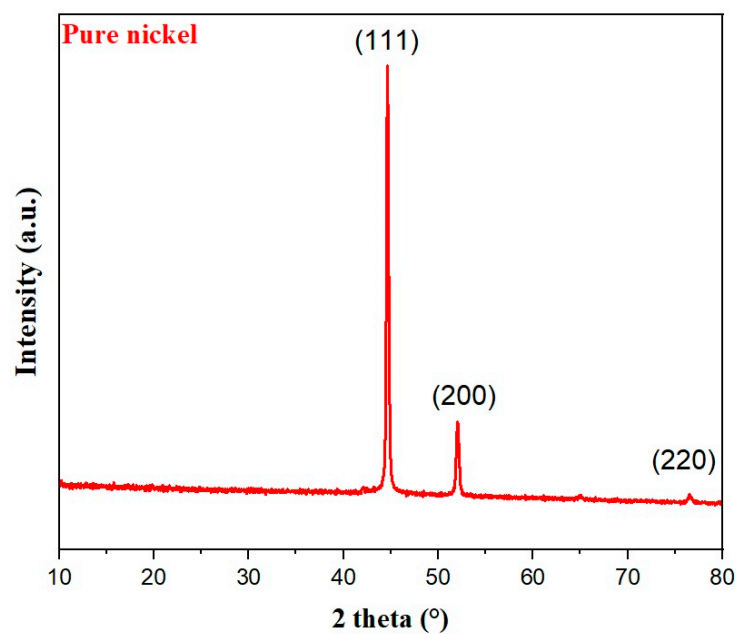


Figure 2. XRD pattern of the nickel coating.

In the Ni-Al₂O₃ composite coatings, the same characteristic peaks of nickel are observed, confirming the dominant presence of the nickel phase in the coating. However, in the XRD diffractograms, shown in Figure 3, the peaks exhibit a switched preferential orientation to the (200) plane with a slight broadening and increased intensity compared to pure nickel, which can be attributed to the incorporation of Al₂O₃ particles, leading to a refinement of the crystallite size and potential lattice strain [22]. The absence of distinct peaks corresponding to Al₂O₃ suggests its uniform dispersion within the nickel matrix, possibly in an amorphous or nanocrystalline state [26].

We conducted a detailed investigation into the impact of electrodeposition time on crystal growth and grain orientation in coatings formed under varying current densities. Notably, the current density exhibited a significant influence on the growth process and grain orientation. Specifically, the diffraction peak intensity of the (111) plane diminished progressively, while that of the (200) plane increased correspondingly. Furthermore, the texture orientation of the coating transitioned from the (200) to the (111) plane as hydrogen coverage increased. This phenomenon may be attributed to the degree of the hydrogen evolution reaction or the incorporation of adsorbed hydrogen (H_{ads}) into the deposit [10]. The electrodeposition mechanism of nickel (Ni) is described by Reactions (4)–(6) [27–30]:



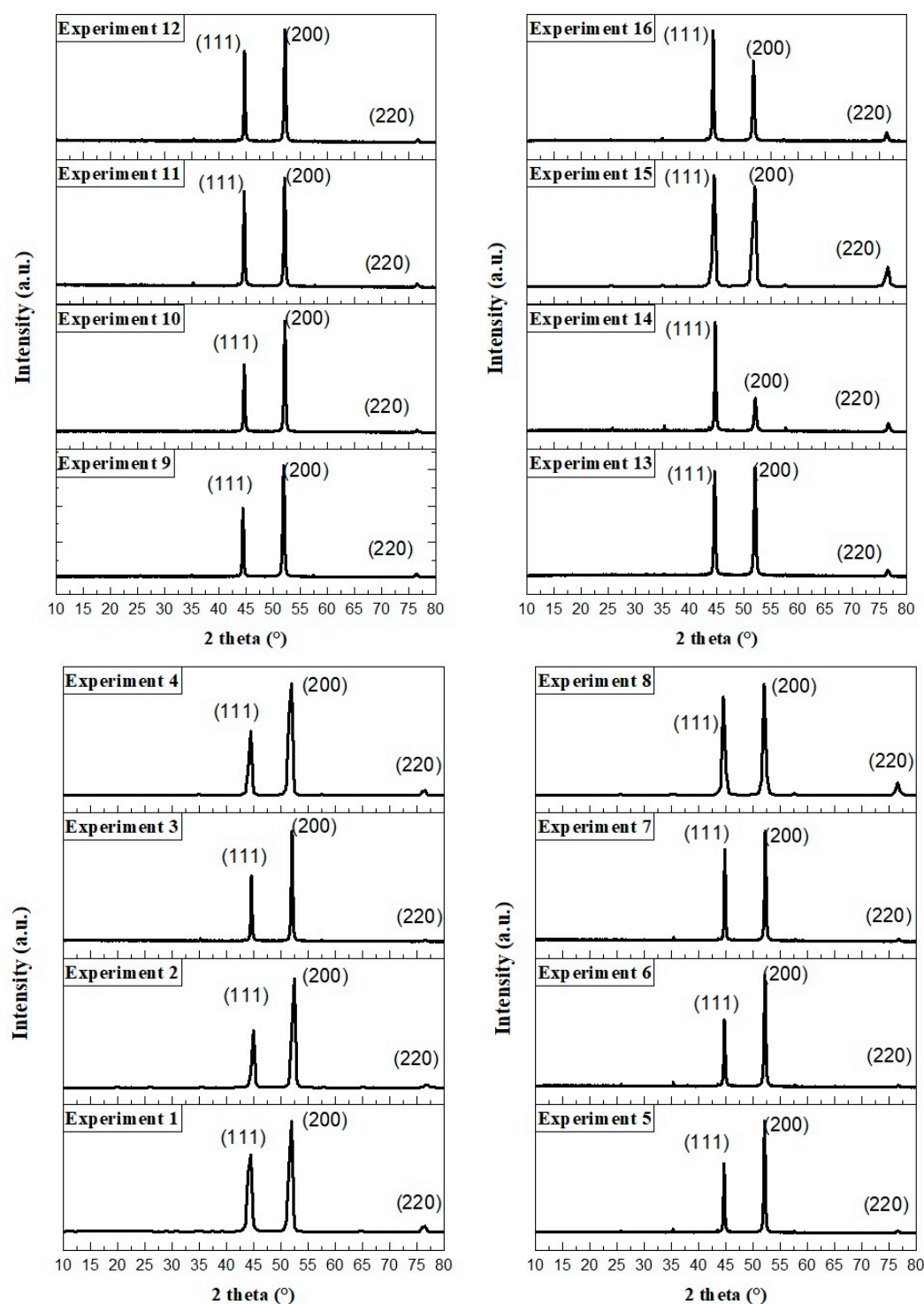


Figure 3. XRD pattern of the 16 fabricated Ni-Al₂O₃ composite coatings, identified according to the design of the experiment plan presented in Table 3.

From Equations (4)–(6) and according to the above interpretation, it can be inferred that the X^- anion is hydroxide (OH^-). The variation in deposit texture orientation may arise from inhibitors such as H_2 , H_{ads} , $\text{Ni}(\text{OH})_2$, or $\text{Ni-H}_3\text{BO}_3$ complexes at the deposit–electrolyte interface [10].

Regarding the co-deposition of alumina, the typical process for co-depositing nanoparticles into growing metallic layers consists of five main stages: the formation of ionic clouds around the particles, their movement towards the cathode via convection, diffusion through the hydrodynamic boundary layer, diffusion across the concentration boundary layer, and,

ultimately, adsorption at the cathode, where the nanoparticles are embedded within the metal deposit [31].

The orientation of Ni crystallites is influenced by alumina incorporation, deposition time, and current density, significantly impacting anisotropic mechanical properties.

3.2. SEM Examination

The displayed SEM images illustrate the surface morphology of pure nickel and Ni-Al₂O₃ coatings electrodeposited at a current density of 2 A/dm² for 30 min. As shown in Figure 4a, the micrograph reveals a uniform, dense, and reasonably homogeneous surface coating structure, indicative of effective deposition under the specified parameters. The surface appears nodular with fine granular features, characteristic of high-purity nickel deposition. Additionally, the absence of visible porosity is evident, as no pinholes or other defects are observed.

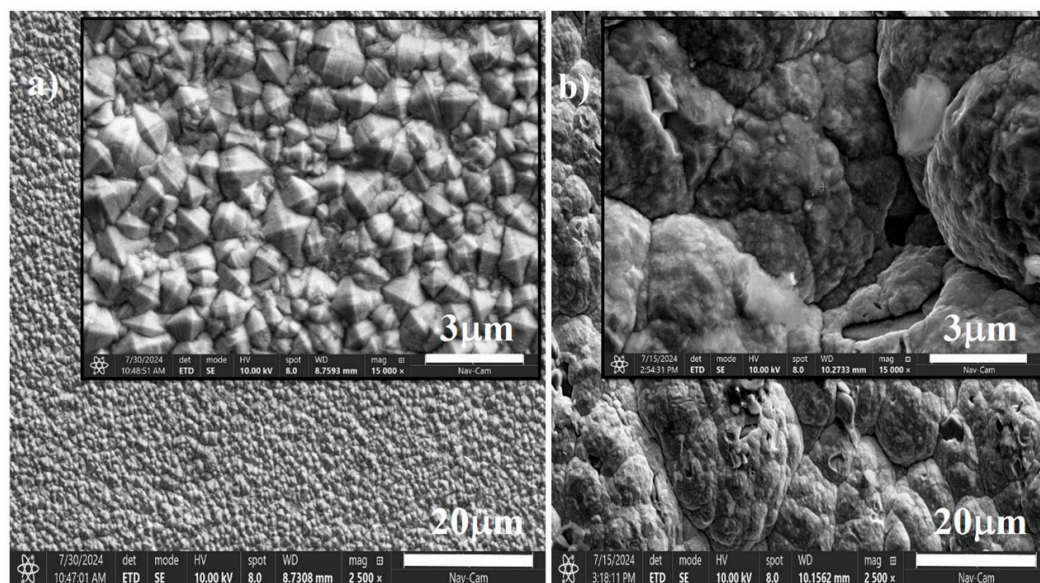


Figure 4. SEM micrographs of the surface of (a) pure Ni coating and (b) Ni-Al₂O₃ composite coating.

Figure 4b provides SEM micrographs of the electrodeposited Ni-Al₂O₃ composite coating, resulting from the addition of alumina particles to the chloride bath. The coating exhibits a densely packed, granular structure with a uniform distribution of alumina particles throughout the nickel matrix. A distinct heterogeneity is observed, with signs of low compactness and rounded clusters resembling a cauliflower-like surface morphology. This contrasts with the typical pyramid-like structures found in pure nickel coatings. The incorporation of alumina particles appears to disrupt the usual growth pattern of nickel grains, transforming them from angular, pyramid-like formations to a more globular, cauliflower-like structure. This change is likely due to alumina particles acting as nucleation sites, promoting isotropic growth and leading to the observed granular morphology. This structure can enhance the coating's surface area and improve properties such as hardness and wear resistance.

This view is further supported by energy-dispersive spectroscopy (EDS) analysis. Figure 5 confirms the successful incorporation of Al₂O₃ particles into the Ni matrix during electrodeposition, as indicated by the intense peaks of Al and O in the spectrum of the Ni-Al₂O₃ composite (Figure 5b). In contrast, Figure 5a shows that the pure Ni coating is predominantly composed of nickel with a low presence of oxygen, which aligns with the XRD analysis. The inset presents the weight and atomic percentages of the elemental composition of the coatings.

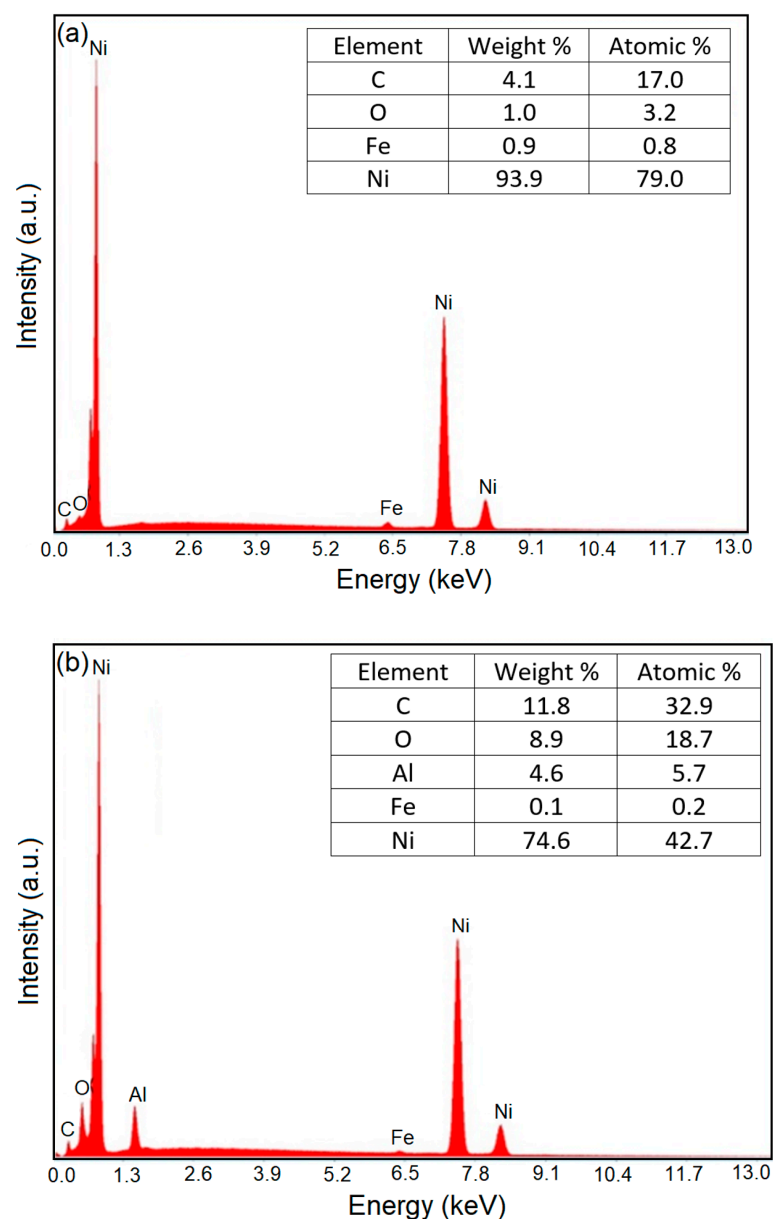


Figure 5. EDS spectra of (a) pure Ni coating and (b) Ni-Al₂O₃ composite coating.

Figure 6 displays SEM/EDS mapping, illustrating the elemental distribution of Ni and Al on the surface of the Ni-Al₂O₃ composite coating. The Al (Ka1) mapping highlights concentrated clusters of alumina particles, while the Ni (Ka1) mapping reveals a more continuous distribution, suggesting that nickel forms the predominant metallic matrix. These clusters exhibit a significantly stronger Al signal due to the agglomeration of alumina nanoparticles. This implies that freshly entrapped alumina nanoparticles were incorporated during electrodeposition, likely due to their high surface activity [32–34], with Ni preferentially depositing around them. Similar findings were reported in [35], where it was concluded that the presence of Al₂O₃ nanoparticles obstructs the movement of dislocations in the nickel matrix, yielding an increase in grain size [32], which, in turn, enhances hardness and corrosion resistance [36].

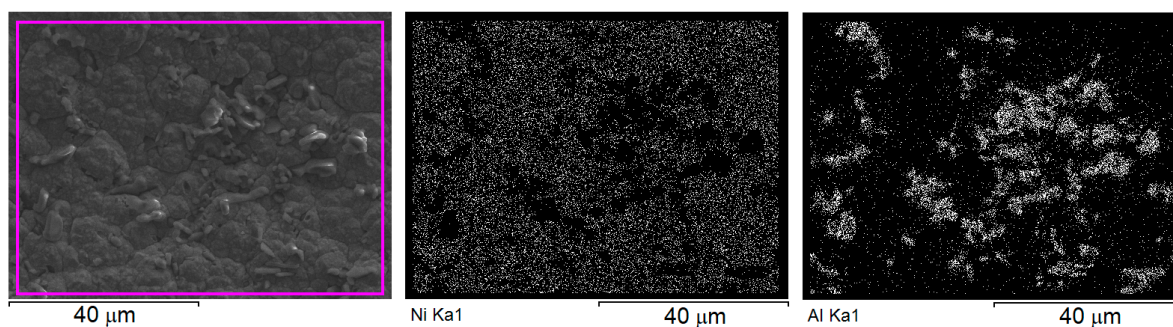


Figure 6. SEM/EDS mapping showing the elemental distribution on the surface of the Ni-Al₂O₃ composite coating.

3.3. Analysis of Variance

The Taguchi L16 analysis plan and results for the electrodeposition process are presented in Tables 4 and 5, highlighting the effect of the process parameters on the coating's microhardness, average crystallite size, and incorporated alumina content.

Table 4. Coded and uncoded formulation of electrodeposition input parameters, current density, alumina concentration, and deposition time, according to the Taguchi L16 analysis plan.

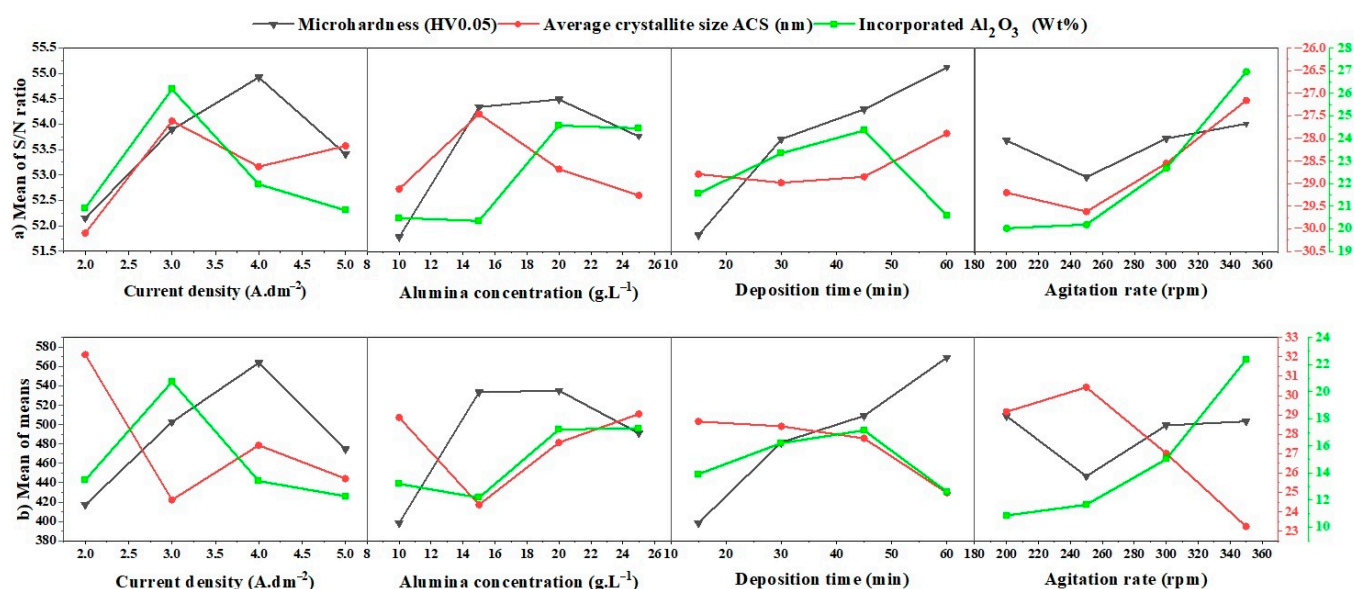
Run	Coded Matrix				Current Density (A.dm ⁻²)	Alumina Concentration (g.L ⁻¹)	Deposition Time (min)	Agitation Rate (rpm)
	X1	X2	X3	X4				
1	1	1	1	1	2	10	15	200
2	1	2	2	2	2	15	30	250
3	1	3	3	3	2	20	45	300
4	1	4	4	4	2	25	60	350
5	2	1	2	3	3	10	30	300
6	2	2	1	4	3	15	15	350
7	2	3	4	1	3	20	60	200
8	2	4	3	2	3	25	45	250
9	3	1	3	4	4	10	45	350
10	3	2	4	3	4	15	60	300
11	3	3	1	2	4	20	15	250
12	3	4	2	1	4	25	30	200
13	4	1	4	2	5	10	60	250
14	4	2	3	1	5	15	45	200
15	4	3	2	4	5	20	30	350
16	4	4	1	3	5	25	15	300

The microhardness values obtained in this study demonstrated a significant increase of over 164%, rising from 269 HV (Run 1) to 712 HV (Run 10). This enhancement surpasses the typical increase of approximately 80% reported by several researchers [37–39]. Additionally, the average crystallite size decreased from 36 nm (Run 1) to 17 nm (Run 6), indicating an improvement of around 400%. These values are smaller than those reported in the literature [40]. Moreover, the weight percentage of incorporated Al₂O₃ in the coatings ranged from 5% (Run 1) to 25% (Run 6), which is higher than the values typically observed in the literature, where the wt% of alumina usually falls between 5% and 16% [39].

Figure 7 and Tables 6–8 clearly illustrate the significant impact of the average signal-to-noise ratio (S/N) on the material properties under investigation. The findings suggest that higher S/N ratios correspond to notable improvements in these responses.

Table 5. Microhardness, average crystallite size, and incorporated alumina content of the coatings, along with the corresponding signal-to-noise (S/N) ratio, according to the Taguchi L16 analysis plan.

Run	Coded Matrix				Microhardness (HV _{0.05})	S/N Ratio (dB)	Average Crystallite Size (nm)	S/N Ratio (dB)	Incorporated Alumina (wt%)	S/N Ratio (dB)
	X1	X2	X3	X4						
1	1	1	1	1	269.00	48.5950	36.0	−31.1261	5.0	13.9794
2	1	2	2	2	415.38	52.3689	33.0	−30.3703	7.0	16.9020
3	1	3	3	3	468.75	53.4188	30.4	−29.6653	18.7	25.4368
4	1	4	4	4	515.00	54.2361	29.0	−29.2480	23.2	27.3135
5	2	1	2	3	416.00	52.3819	28.5	−29.1017	22.6	27.0816
6	2	2	1	4	446.10	52.9886	16.8	−24.5183	25.0	27.9684
7	2	3	4	1	652.71	56.2944	24.0	−27.6056	14.9	23.4675
8	2	4	3	2	496.00	53.9096	29.1	−29.2714	20.4	26.1790
9	3	1	3	4	511.10	54.1701	25.0	−27.9638	19.6	25.8441
10	3	2	4	3	712.00	57.0496	21.0	−26.4400	6.8	16.5874
11	3	3	1	2	478.00	53.5886	33.7	−30.5576	12.3	22.6978
12	3	4	2	1	553.57	54.8635	30.0	−29.5424	13.6	22.6978
13	4	1	4	2	397.00	51.9758	25.9	−28.2795	5.6	14.9974
14	4	2	3	1	560.00	54.9638	26.6	−28.5096	9.9	19.9510
15	4	3	2	4	541.00	54.6639	22.1	−26.9038	21.6	26.6792
16	4	4	1	3	400.00	52.0412	28.1	−28.9778	12.0	21.5962

**Figure 7.** Taguchi design results for the microhardness, ACS, and Wt%(Al₂O₃). (a) Main effect plot of S/N ratio, (b) Main effect of means.**Table 6.** Response table of mean S/N ratio of microhardness of Ni-Al₂O₃ composite coatings.

Response Variable		Mean S/N Ratio			
Microhardness	Level	Current Density (A.dm ⁻²)	Alumina Concentration (g.L ⁻¹)	Deposition Time (min)	Agitation Rate (rpm)
	1	52.15	51.78	51.80	53.68
	2	53.89	54.34	53.57	52.96
	3	54.92	54.49	54.12	53.72
	4	53.41	53.76	54.89	54.01
Δ		2.76	2.71	3.09	1.05
Rank		2	3	1	4

Table 7. Response table of mean S/N ratio of incorporated amount of alumina in Ni-Al₂O₃ composite coatings.

Response Variable		Mean S/N Ratio			
Incorporated Alumina	Level	Current Density (A.dm ⁻²)	Alumina Concentration (g.L ⁻¹)	Deposition Time (min)	Agitation Rate (rpm)
	1	20.91	20.48	21.56	20.02
	2	26.17	20.35	23.34	20.19
	3	21.96	24.57	24.35	22.68
	4	20.81	24.45	20.59	26.95
Δ		5.37	4.22	3.76	6.93
Rank		2	3	4	1

Table 8. Response table of mean S/N ratio of ACS of Ni-Al₂O₃ composite coatings.

Response Variable		Mean S/N Ratio			
Average Crystallite Size—ACS	Level	Current Density (A.dm ⁻²)	Alumina Concentration (g.L ⁻¹)	Deposition Time(min)	Agitation Rate (rpm)
	1	−30.10	−29.12	−28.79	−29.20
	2	−27.62	−27.46	−28.98	−29.62
	3	−28.63	−28.68	−28.85	−28.55
	4	−28.17	−29.26	−27.89	−27.16
Δ		2.48	1.80	1.09	2.6
Rank		1	3	4	2

Regarding the effect of parameters A, B, C, and D on microhardness (Table 6), the S/N ratio model analysis reveals significant effects from all parameters, except for (D) the agitation rate. Parameters (A) current density, (C) alumina concentration, and (B) deposition time show high significance. Among these, deposition time has the largest influence, followed by alumina concentration and current density, with contributions of 35.50%, 32.24%, 27.01%, and 5.03%, respectively. This indicates that the parameters tested play crucial roles in optimizing the robustness of the electrodeposition process.

The main effects and ranking show that time deposition ranks highest for both the S/N ratio and the HV average response, followed closely by alumina concentration and current density, while the agitation rate has the least impact. S. Pouladi et al. [41] stated that variations in the agitation rate have very little effect on the coatings' microhardness. Higher hardness values and reliable results can only be obtained by adjusting the deposition time and alumina concentration, according to this ranking. A similar arrangement was reported by S.T. Aruna [5] using the Taguchi L9 technique. The evaluation of average Vickers hardness also revealed consistent patterns, with current density, particle concentration, and electrodeposition time having a significant impact, while the agitation rate had a negligible effect. These findings, consistent with the signal-to-noise ratio study, show that variations in agitation rate have only a minimal effect on increasing the composite coating's microhardness.

Regarding the weight percentage of incorporated alumina (Table 7), the analysis highlights significant contributions from all four factors, with agitation rate having the most pronounced influence. The mean of the mean response shows a clear hierarchy, with agitation rate ranking first, followed closely by current density, alumina concentration, and deposition time. This trend confirms the dominance of agitation rate and current density as key factors in achieving higher average responses. In [42], the study investigated the

hydrodynamic effect on the incorporation rate, identifying an optimal rate influenced by both particle concentration and agitation rate.

The significant effect of alumina concentration, although lower than the agitation rate and current density effects, underscores its supportive role, while deposition time remains the least impactful. These findings align well with the S/N ratio analysis and suggest that prioritizing agitation rate and applied current density (with 38.11% and 23.38%, respectively) adjustments is critical for optimizing mean response values. Alumina concentration and deposition time offer additional, though less substantial, enhancement.

For the average crystallite size (Table 8), the analysis elucidates that agitation rate is the most influential factor across both signal-to-noise ratios and mean values, contributing 32.81% to the overall variability, followed by current density with 32.07%. Alumina concentration and deposition time contribute 18.97% and 7.01%, respectively, indicating their lower impact. The ranking of effects for the mean response similarly places current density first, followed closely by agitation rate, alumina concentration, and deposition time. This ranking confirms and supports the previous research [35,41,43]. The significance of current density at a certain level highlights its relevance. Agitation rate, though slightly less dominant, also plays a central role.

3.4. Linear Regression

Multiple linear regression models were developed using Design-Expert13 software to analyze the responses of the microhardness (Y_1), the alumina content (Y_2), and the average crystallite size (Y_3):

$$Y_1 = 560.064 + 81.0518 \times A + 42.4297 \times B + 136.881 \times C + 21.9426 \times D + 107.932 \times AB + 8.05077 \times AC - 33.3965 \times AD + 74.6297 \times BC - 78.681 \times BD + 44.0988 \times CD - 30.6895 \times A^2 + -122.775 \times B^2 + 26.4045 \times C^2$$

$$Y_2 = 14.9752 + 2.34727 \times A + 0.00674076 \times B - 3.68038 \times C + 3.76151 \times D - 5.52969 \times AB - 4.39055 \times AC + 0.299968 \times AD + 8.85034 \times BC - 4.58164 \times BD - 2.69986 \times CD$$

$$Y_3 = 27.4626 - 3.18263 \times A - 2.62645 \times B - 4.30298 \times C - 5.43546 \times D - 5.28756 \times AB - 5.83117 \times AC + 2.32292 \times AD - 3.59736 \times BC + 1.41072 \times BD + 4.66367 \times CD$$

As can be seen from Table 9, the interaction of current density and alumina concentration has the highest influence on the microhardness of the fabricated coating. Also, it shows that, in the incorporated amount of alumina, the interaction of alumina concentration and deposition time (BC) has the highest influence, followed by the interaction of agitation rate and the alumina concentration (BD). Finally, the unique factor interactions highly influencing the ACS of the Ni-Al₂O₃ composite coating are deposition time and agitation rate.

Table 9. ANOVA for the linear model of microhardness, weight percentage of incorporated alumina, and average crystallite size of the composite coating.

Source		R ²	DF	Adj SS	Adj MS	F-Value	p-Value
Microhardness	Model	99.7	13	1.672 × 10 ⁵	12,860.80	69.70	0.0142
	A		1	14,956.40	14,956.40	81.06	0.0121
	B		1	3881.85	3881.85	21.04	0.0444
	C		1	41,722.50	41,722.50	226.12	0.0044
	D		1	1999.95	1999.95	10.84	0.0812
	AB		1	8637.51	8637.51	46.81	0.0207
	AC		1	62.32	62.32	0.3378	0.6199
	AD		1	2299.25	2299.25	12.46	0.0717

Table 9. Cont.

	Source	R ²	DF	Adj SS	Adj MS	F-Value	p-Value
Incorporated alumina (Wt %)	BC		1	4400.66	4400.66	23.85	0.0395
	BD		1	2153.30	2153.30	11.67	0.0760
	CD		1	5657.31	5657.31	30.66	0.0311
	A ²		1	309.71	309.71	1.68	0.3245
	B ²		1	37,464.64	37,464.64	203.04	0.0049
	C ²		1	673.52	673.52	3.65	0.1962
	D ²		0	0.0000			
	Residual		2	369.03	184.52		
	Total		15	1.676 × 10 ⁵			
	Model	92.8	10	641.11	64.11	6.46	0.0263
	A		1	16.08	16.08	1.62	0.2590
	B		1	0.0001	0.0001	9.874 × 10 ^{−6}	0.9976
	C		1	30.16	30.16	3.04	0.1417
	D		1	58.77	58.77	5.92	0.0591
	AB		1	22.67	22.67	2.28	0.1910
	AC		1	18.54	18.54	1.87	0.2300
	AD		1	0.1855	0.1855	0.0187	0.8966
	BC		1	123.68	123.68	12.46	0.0167
	BD		1	84.35	84.35	8.50	0.0332
	CD		1	28.93	28.93	2.92	0.1485
	Residual		5	9.92			
	Total		15				
Average crystallite size	Model	92.5	10	341.30	34.13	6.20	0.0287
	A		1	29.56	29.56	5.37	0.0682
	B		1	14.87	14.87	2.70	0.1610
	C		1	41.23	41.23	7.50	0.0409
	D		1	122.72	122.72	22.31	0.0052
	AB		1	20.73	20.73	3.77	0.1099
	AC		1	32.70	32,70	5.94	0.0588
	AD		1	11.12	11.12	2.02	0.2143
	BC		1	20.43	20.43	3.71	0.1119
	BD		1	8.00	8.00	1.45	0.2819
	CD		1	86.32	86.32	15.69	0.0107
	Residual		5	27.50	5.50		
	Total		15	368.81			

Furthermore, the models were validated by high correlation coefficients, ensuring their robustness in identifying the most influential factors affecting the measured responses.

3.5. Three-Dimensional Response Surface Approach Analysis

The fabrication of Ni-Al₂O₃ electrodeposited composite coatings was optimized through 3D surface analysis using Design-Expert13 software. This approach enabled a comprehensive evaluation of the interactions between key input variables and their effects on the coating's properties. The 3D surface plots in Figures 8–10 visually represent the responses in relation to the microhardness, average crystallite size, and incorporated alumina with the combined influence of parameter interactions (AB, AC, AD, BC, BD, and CD). The 3D surface allows two variables to be analyzed simultaneously while the other two variables remain in the optimum response target. Strong agreement between experimental and anticipated values demonstrates the excellent predictive accuracy of

the Design-Expert models, underscoring the efficiency of this optimization technique in fine-tuning process parameters for optimal material properties.

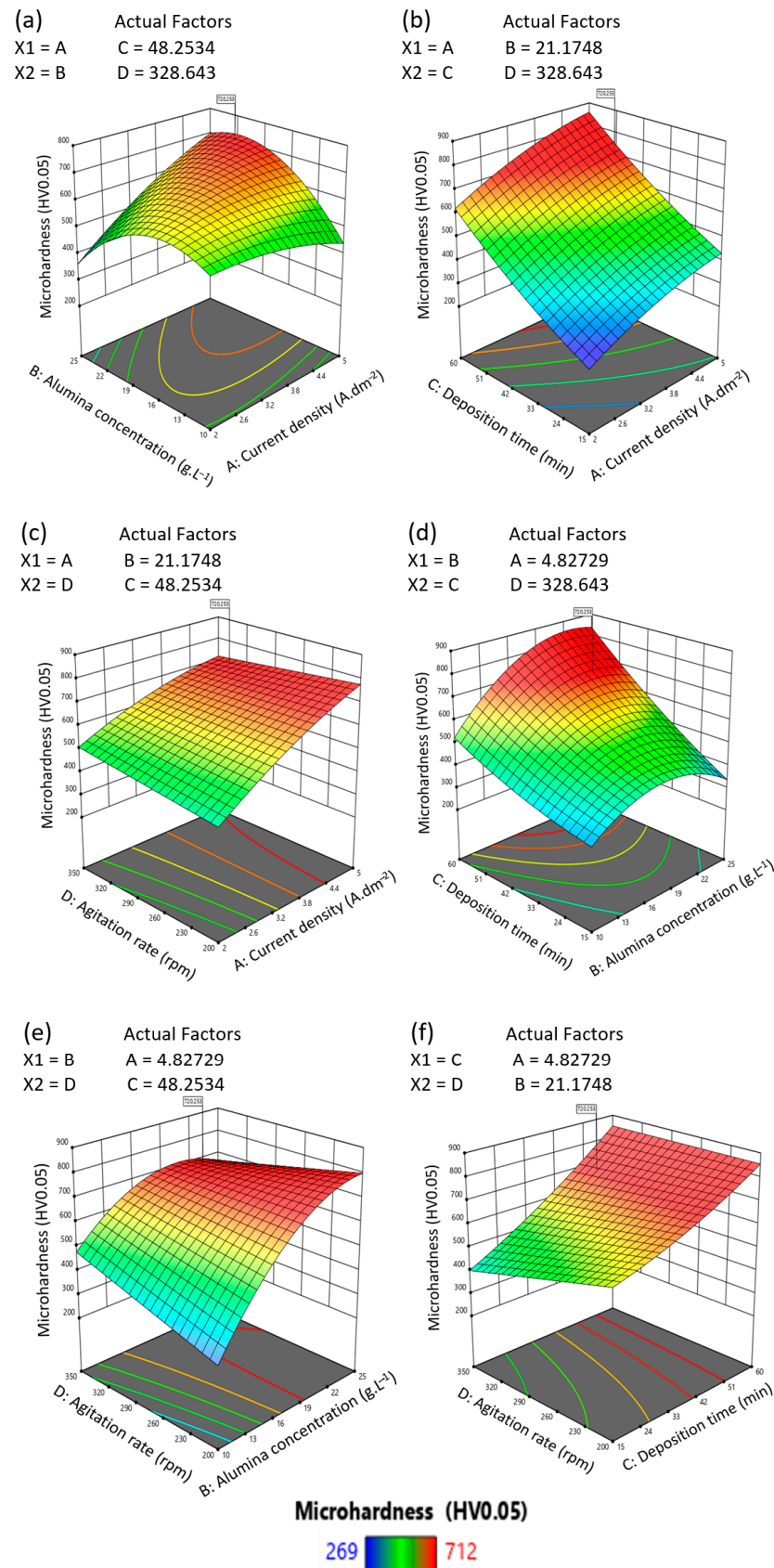


Figure 8. Three-dimensional plots depicting the impact of input parameters and their interactions on microhardness.

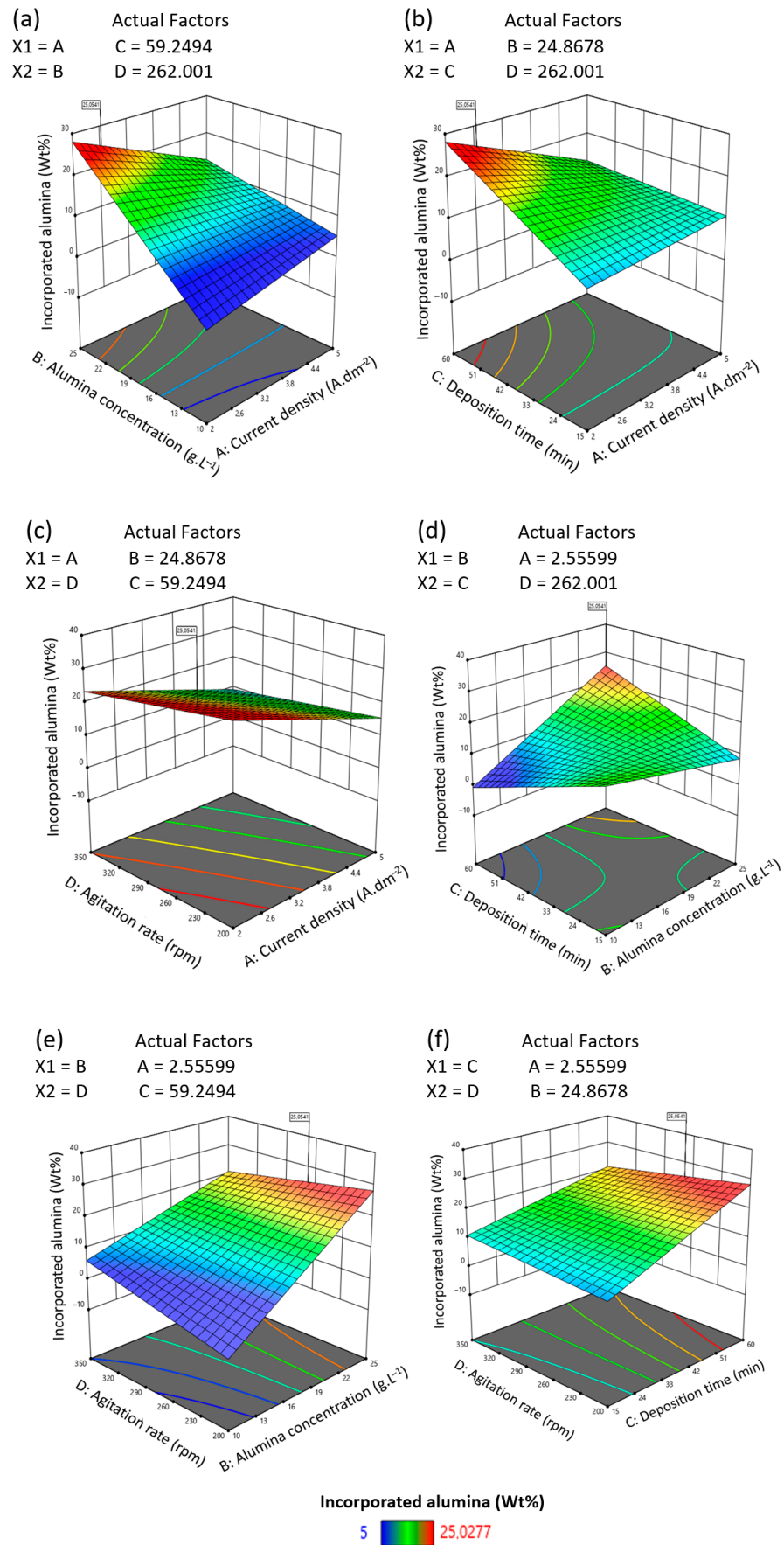


Figure 9. Three-dimensional plots depicting the impact of input parameters and their interactions on the incorporated amount of Al_2O_3 in the coating.

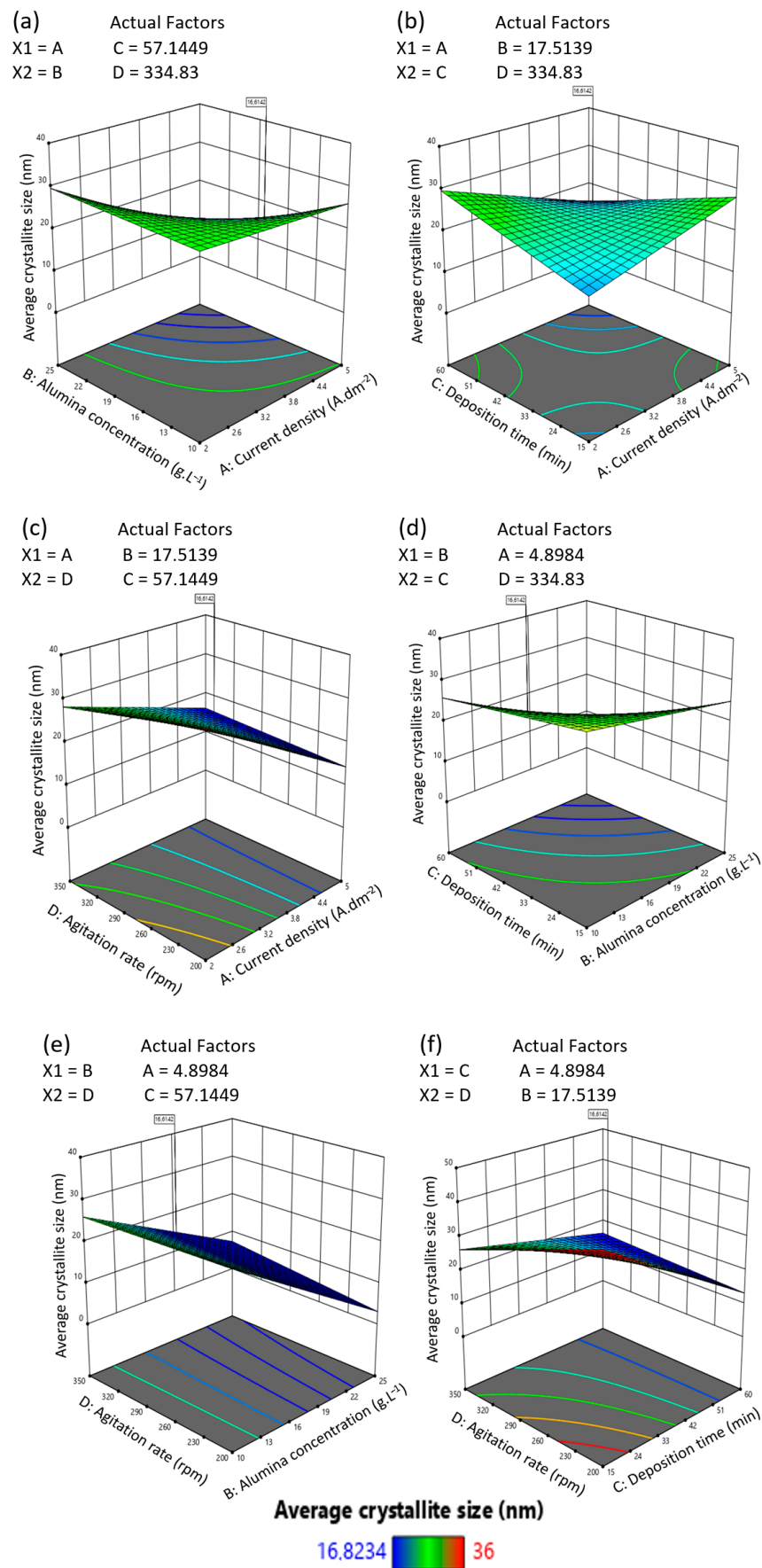


Figure 10. Three-dimensional plots depicting the impact of input parameters and their interactions on the average crystallite size of the composite coating.

A probability diagram is a graphical representation used to show the distribution of random variables or the possible outcomes of different events, commonly utilized in fields such as statistics, mathematics, and economics. Clearer insights into links and variations within a dataset are made possible by these diagrams, which are especially useful when comparing observed data with mathematical forecasts. Figure 11 exhibits a close alignment with the straight line, suggesting the data's validity.

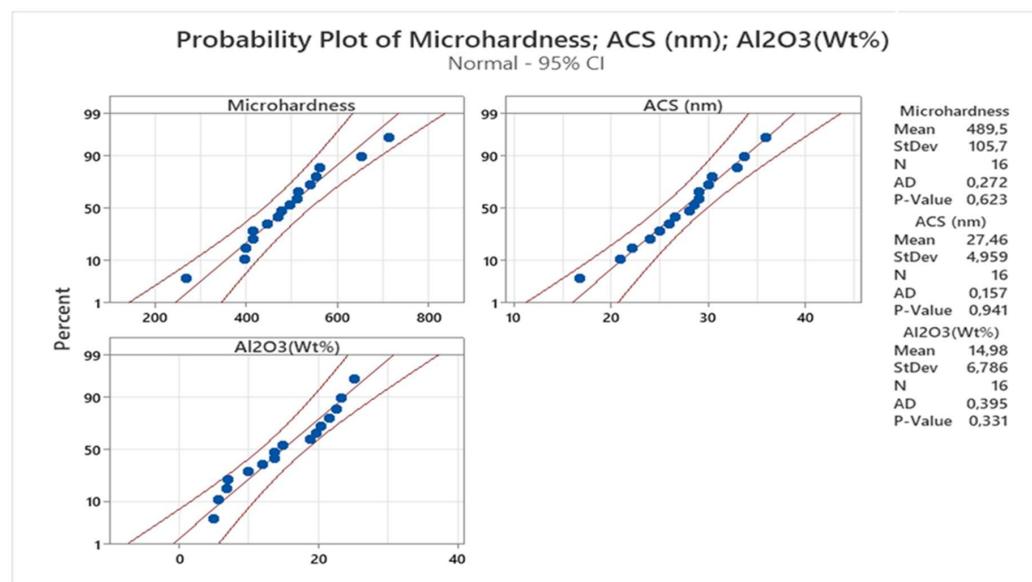


Figure 11. Probability plot using Minitab (Version 21.2.0.0) for microhardness, ACS, and Wt% of alumina.

A p -value of less than 0.05 indicates a statistically significant deviation from normality, thereby necessitating the rejection of the null hypothesis that the data are normally distributed. Conversely, p -values greater than 0.05 imply no significant deviation from normality [13,39].

Our analysis resulted in the following calculated p -values: 0.623, 0.331, and 0.941, for microhardness, weight percentage of Al_2O_3 , and average crystallite size, respectively. Likewise, the highest allowable chance of departure from normalcy is restricted to 5% within a 95% confidence range, emphasizing the statistical rigor used in these evaluations.

3.6. Optimization

We conducted linear regression to fine-tune the parameters for co-electrodepositing Ni- Al_2O_3 composite coatings. Our goal was to maximize microhardness and alumina content while minimizing crystallite size. To achieve this, we focused on four key factors: the applied current density (A), alumina concentration in the bath (B), deposition time (C), and agitation rate (D).

The optimized parameter combination provided a high desirability value, close to 1, indicating an excellent balance of outcomes [44,45]. The desirability function was crucial in identifying the best set of parameters, ensuring optimal results among the various possibilities suggested by the software.

Figure 12 highlights the optimal response values achieved: a microhardness of 762, an average crystallite size of 15.36 nm, and 29.09% Al_2O_3 incorporation. These results were obtained using an applied current density of 4.0606 A/dm^2 , an alumina concentration of 20.4545 g/L , a deposition time of 60 min, and an agitation rate of 340.9091 rpm, culminating in a composite desirability value of 1.

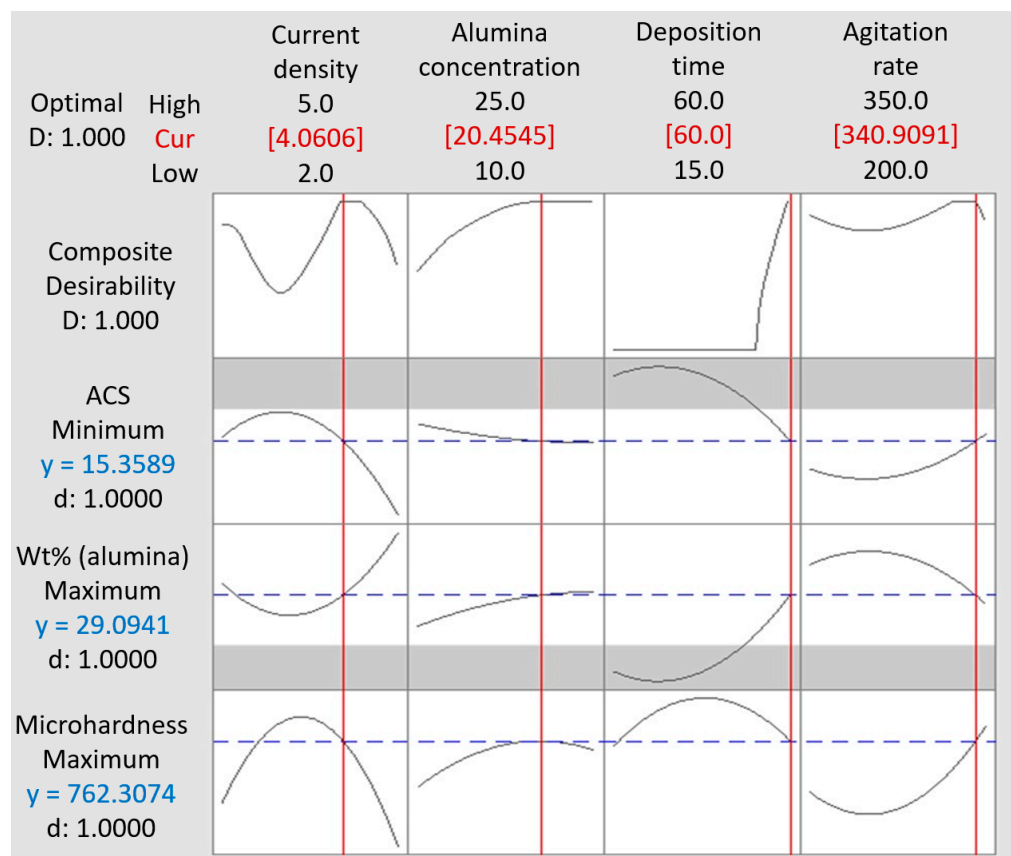


Figure 12. Response optimization plot for electrodeposited Ni-Al₂O₃ composite coating.

The identified desirability score of 1 presents the ideal solution over the values in the considered ranges, identified in Figure 12, where all the objectives are optimized simultaneously.

The optimal obtained results are supported by [9], demonstrating higher microhardness, finer crystallite size, and comparable optimal particle concentration in the bath.

The predictive models developed using Taguchi's method showed a strong match between experimental and predicted results. This confirms their accuracy and highlights their competitiveness compared to other popular modeling approaches like artificial neural networks (ANN) and fuzzy logic methods, which have also been widely used to model Ni-Al₂O₃ electrodeposited composite coatings, namely, in the reported investigations of [46,47].

4. Conclusions

In this study, the Taguchi L16 orthogonal array was employed to optimize the microhardness, crystallite size, and entrapped alumina content of Ni-Al₂O₃ electrodeposited coatings.

The key process parameters, current density, electrodeposition time, alumina concentration, and agitation rate, were systematically varied to determine their optimal levels, with the experimental results being analyzed and validated using ANOVA to ensure statistical reliability.

The coatings developed exhibited remarkable improvements, including a 164% increase in microhardness, a 400% reduction in crystallite size, and a higher alumina incorporation (5–25%) compared to typical reported values.

The developed models demonstrated strong predictive performance, as indicated by the R² and R²-predicted values of 92% and 99%, respectively. These results underscore the reliability of the models in accurately predicting responses, enabling process optimization that leads to substantial reductions in processing time and material consumption. Such

advancements contribute to enhanced cost efficiency, improved coating performance, and greater sustainability in industrial applications, making electrodeposited Ni-Al₂O₃ coatings a more viable and competitive solution for wear-resistant and corrosion-resistant applications.

Author Contributions: L.G.: resources and supervision. S.T.: supervision and validation. I.R.: conceptualization and writing—original draft and editing. F.L.: methodology and investigation. P.H.: data curation, methodology, and investigation. S.D.: revising. H.B.: validation. All authors have read and agreed to the published version of the manuscript.

Funding: The work is financed by the Algerian Ministry of Higher Education and Scientific Research (MESRS) and also through the FCT—Fundação para a Ciência e Tecnologia, under projects UIDB/00285—Centre for Mechanical Engineering, Materials and Processes and LA/P/0112/2020.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Sajjadnejad, M.; Haghshenas, S.M.S.; Badr, P.; Setoudeh, N.; Hosseinpour, S. Wear and Tribological Characterization of Nickel Matrix Electrodeposited Composites: A Review. *Wear* **2021**, *486–487*, 204098. [\[CrossRef\]](#)
2. Singh, S.; Kumar, S.; Khanna, V. A Review on Surface Modification Techniques. *Mater. Today Proc.* **2023**; in press. [\[CrossRef\]](#)
3. Holden, K.E.K.; Qi, Y.; Conley, J.F. Precision Defect Engineering of Metal/Insulator/Metal Diodes Using Atomic Layer Deposition to Localize Ni Impurities in Al₂O₃ Tunnel Barriers. *J. Appl. Phys.* **2021**, *129*, 144502. [\[CrossRef\]](#)
4. Kim, M.; Kang, K.-M.; Wang, Y.; Park, H.-H. N-Doped Al₂O₃ Thin Films Deposited by Atomic Layer Deposition. *Thin Solid Films* **2018**, *660*, 657–662. [\[CrossRef\]](#)
5. Aruna, S.T.; Srikanth, P.V.K.; Ahamad, M.J.; Latha, S.; Rajam, K.S. Optimization of the Properties of Electrodeposited Ni-YSZ Composites Using Taguchi Method and Regression Analysis. *Port. Electrochim. Acta* **2011**, *29*, 23–37. [\[CrossRef\]](#)
6. Abdesselam, Y.; Rezgui, I.; Naoun, M.; Belloufi, A.; Mezoudj, M.; Zerrouki, D. Experimental Investigation and Optimization of Manufacturing Processes of Ni-P-Y₂O₃ Composite Coatings by Multiple Linear Regression Method Based on Genetic Algorithm. *Int. J. Adv. Manuf. Technol.* **2023**, *126*, 3995–4019. [\[CrossRef\]](#)
7. Wasekar, N.P.; Haridoss, P.; Seshadri, S.K.; Sundararajan, G. Influence of Mode of Electrodeposition, Current Density and Saccharin on the Microstructure and Hardness of Electrodeposited Nanocrystalline Nickel Coatings. *Surf. Coat. Technol.* **2016**, *291*, 130–140. [\[CrossRef\]](#)
8. Mohanty, U.S.; Tripathy, B.C.; Singh, P.; Keshavarz, A.; Iglauer, S. Roles of Organic and Inorganic Additives on the Surface Quality, Morphology, and Polarization Behavior during Nickel Electrodeposition from Various Baths: A Review. *J. Appl. Electrochem.* **2019**, *49*, 847–870. [\[CrossRef\]](#)
9. Saied, S.; Mekkaoui, A.; Belahssen, O.; Chala, A. Effect of TiO₂ nanoparticles in Ni matrix on mechanical and corrosion resistance properties. *Acta Metall. Slovaca* **2017**, *23*, 37–44. [\[CrossRef\]](#)
10. Wu, W.; Huang, J.; Näther, J.; Omar, N.A.B.; Köster, F.; Lampke, T.; Liu, Y.; Pan, H.; Zhang, Y. Texture Orientation, Morphology and Performance of Nanocrystalline Nickel Coatings Electrodeposited from a Watts-Type Bath: Effects of H₃BO₃ Concentration and Plating Time. *Surf. Coatings Technol.* **2021**, *424*, 127648. [\[CrossRef\]](#)
11. Raghavendra, C.R.; Basavarajappa, S.; Sogalad, I. Sliding Wear Behaviour of Ni- α -Al₂O₃ Nano Composite Coating at Elevated Temperatures. *Colloid Interface Sci. Commun.* **2018**, *27*, 18–25. [\[CrossRef\]](#)
12. Karthik, R.; Mani, R.; Manikandan, P. Tribological Studies of Ni-SiC and Ni-Al₂O₃ Composite Coatings by Pulsed Electrodeposition. *Mater. Today Proc.* **2021**, *37*, 701–706. [\[CrossRef\]](#)
13. Benamrane, R.; Bennouna, M.S.; Fellah, M.; Sadek, K. The Improvement of the Tensile Properties of Alfa Fibers Using the Taguchi Method. *Ind. Crops Prod.* **2024**, *221*, 119398. [\[CrossRef\]](#)
14. Kumar, S.; Prasad, L.; Kumar, S.; Patel, V.K. Physico-Mechanical and Taguchi-Designed Sliding Wear Properties of Himalayan Agave Fiber Reinforced Polyester Composite. *J. Mater. Res. Technol.* **2019**, *8*, 3662–3671. [\[CrossRef\]](#)
15. Nguyen, P.H.; Banh, T.L.; Mashood, K.A.; Tran, D.Q.; Dong Pham, V.; Muthuramalingam, T.; Duc Nguyen, V.; Nguyen, D.T. Application of TGRA-Based Optimisation for Machinability of High-Chromium Tool Steel in the EDM Process. *Arab. J. Sci. Eng.* **2020**, *45*, 5555–5562. [\[CrossRef\]](#)

16. Asif, M.; Tariq, A.; Singh, K.M. Estimation of Thermal Contact Conductance Using Transient Approach with Inverse Heat Conduction Problem. *Heat Mass Transf.* **2019**, *55*, 3243–3264. [\[CrossRef\]](#)
17. Badkar, D.S.; Pandey, K.S.; Buvanashakaran, G. Parameter Optimization of Laser Transformation Hardening by Using Taguchi Method and Utility Concept. *Int. J. Adv. Manuf. Technol.* **2011**, *52*, 1067–1077. [\[CrossRef\]](#)
18. Touati, S.; Ghelani, L.; Zemmouri, A.; Boumediri, H. Optimization of Gas Carburizing Treatment Parameters of Low Carbon Steel Using Taguchi and Grey Relational Analysis (TA-GRA). *Int. J. Adv. Manuf. Technol.* **2022**, *120*, 7937–7949. [\[CrossRef\]](#)
19. Doumi, A.; Rezgui, I.; Belloufi, A.; Abdelkrim, M. Improvement of the manufacturing of Ni-P-Al₂O₃ composite coatings by optimizing the parameters of electroplating process. *Acad. J. Manuf. Eng.* **2022**, *20*, 51–65.
20. Abdulkadir, L.N.; Abou-El-Hossein, K.; Abioye, A.M.; Liman, M.M.; Cheng, Y.-C.; Abbas, A.A.S. Process Parameter Selection for Optical Silicon Considering Both Experimental and AE Results Using Taguchi L9 Orthogonal Design. *Int. J. Adv. Manuf. Technol.* **2019**, *103*, 4355–4367. [\[CrossRef\]](#)
21. Laouissi, A.; Nouioua, M.; Yaltese, M.A.; Abderazek, H.; Maouche, H.; Bouhalais, M.L. Machinability Study and ANN-MOALO-Based Multi-Response Optimization during Eco-Friendly Machining of EN-GJL-250 Cast Iron. *Int. J. Adv. Manuf. Technol.* **2021**, *117*, 1179–1192. [\[CrossRef\]](#)
22. Yang, X.; Zhou, Y.; Zhao, M.; Liu, P.; Qi, H.; Yang, Y. Impact of Al Nanoparticles upon the Microstructure and Wear Properties of Ni-Cr-Al Nanocomposite Coatings. *J. Indian Chem. Soc.* **2023**, *100*, 100822. [\[CrossRef\]](#)
23. Ma, C.; Li, H.; Xia, F.; Xiao, Z. Effect of Ultrasonic Power on Microstructure and Properties of Ultrasonic-Assisted Electrodeposited Ni-Al₂O₃ Thin Nanocoatings. *Trans. Indian Inst. Met.* **2022**, *75*, 1669–1679. [\[CrossRef\]](#)
24. Benea, L.; Danaila, E.; Celis, J.-P. Influence of Electro-Co-Deposition Parameters on Nano-TiO₂ Inclusion into Nickel Matrix and Properties Characterization of Nanocomposite Coatings Obtained. *Mater. Sci. Eng. A* **2014**, *610*, 106–115. [\[CrossRef\]](#)
25. Li, B.; Li, X.; Huan, Y.; Xia, W.; Zhang, W. Influence of Alumina Nanoparticles on Microstructure and Properties of Ni-B Composite Coating. *J. Alloy. Compd.* **2018**, *762*, 133–142. [\[CrossRef\]](#)
26. Wagih, A. Synthesis of Nanocrystalline Al₂O₃ Reinforced Al Nanocomposites by High-Energy Mechanical Alloying: Microstructural Evolution and Mechanical Properties. *Trans. Indian Inst. Met.* **2016**, *69*, 851–857. [\[CrossRef\]](#)
27. Okonkwo, B.O.; Jeong, C.; Jang, C. Advances on Cr and Ni Electrodeposition for Industrial Applications—A Review. *Coatings* **2022**, *12*, 1555. [\[CrossRef\]](#)
28. Estrella-Gutiérrez, M.A.; Lizama-Tzec, F.I.; Arés-Muzio, O.; Oskam, G. Influence of a Metallic Nickel Interlayer on the Performance of Solar Absorber Coatings Based on Black Nickel Electrodeposited onto Copper. *Electrochim. Acta* **2016**, *213*, 460–468. [\[CrossRef\]](#)
29. Graff, A.; Barrez, E.; Baranek, P.; Bachet, M.; Bénézech, P. Complexation of Nickel Ions by Boric Acid or (Poly)Borates. *J. Solut. Chem.* **2017**, *46*, 25–43. [\[CrossRef\]](#)
30. Devesa, S.; Benzarti, Z.; Santos, G.; Cavaleiro, D.; Cunha, A.; Santos, J.; Carvalho, S. Enhancing Solar Absorption with Double-Layered Nickel Coatings and WS₂ Nanoparticles on Copper Substrates. *Energies* **2024**, *17*, 3869. [\[CrossRef\]](#)
31. Santos, G.; Benzarti, Z.; Cavaleiro, D.; Figueiredo, L.; Carvalho, S.; Devesa, S. Optimization of Black Nickel Coatings' Electrodeposition onto Steel. *Coatings* **2024**, *14*, 1125. [\[CrossRef\]](#)
32. Wu, G.; Li, N.; Zhou, D.; Mitsuo, K. Electrodeposited Co-Ni-Al₂O₃ Composite Coatings. *Surf. Coat. Technol.* **2004**, *176*, 157–164. [\[CrossRef\]](#)
33. Jiang, Q.; Lu, H.M. Size Dependent Interface Energy and Its Applications. *Surf. Sci. Rep.* **2008**, *63*, 427–464. [\[CrossRef\]](#)
34. Mersagh Dezfuli, S.; Sabzi, M. Deposition of Ceramic Nanocomposite Coatings by Electroplating Process: A Review of Layer-Deposition Mechanisms and Effective Parameters on the Formation of the Coating. *Ceram. Int.* **2019**, *45*, 21835–21842. [\[CrossRef\]](#)
35. Mamaghani, K.R.; Naghib, S.M. The Effect of Stirring Rate on Electrodeposition of Nanocrystalline Nickel Coatings and Their Corrosion Behaviors and Mechanical Characteristics. *Int. J. Electrochem. Sci.* **2017**, *12*, 5023–5035. [\[CrossRef\]](#)
36. Lv, J. Effect of Grain Size on Mechanical Property and Corrosion Resistance of the Ni-Based Alloy 690. *J. Mater. Sci. Technol.* **2018**, *34*, 1685–1691. [\[CrossRef\]](#)
37. Badarulzaman, N.A.; Purwadaria, S.; Mohamad, A.A.; Ahmad, Z.A. The Production of Nickel–Alumina Composite Coating via Electroplating. *Ionics* **2009**, *15*, 603–607. [\[CrossRef\]](#)
38. Gül, H.; Uysal, M.; Akbulut, H.; Alp, A. Effect of PC Electrodeposition on the Structure and Tribological Behavior of Ni–Al₂O₃ Nanocomposite Coatings. *Surf. Coat. Technol.* **2014**, *258*, 1202–1211. [\[CrossRef\]](#)
39. Borkar, T.; Harimkar, S.P. Effect of Electrodeposition Conditions and Reinforcement Content on Microstructure and Tribological Properties of Nickel Composite Coatings. *Surf. Coat. Technol.* **2011**, *205*, 4124–4134. [\[CrossRef\]](#)
40. Noroozi, Z.; Rajabi, M.; Bostani, B. Characterization of Co-Electrodeposited Functionally Gradient Ni-Al₂O₃ Composite Coating. *J. Surf. Investig. X-Ray Synchrotron Neutron Tech.* **2019**, *13*, 771–776. [\[CrossRef\]](#)
41. Pouladi, S.; Shariat, M.H.; Bahrololoom, M.E. Electrodeposition and Characterization of Ni–Zn–P and Ni–Zn–P/Nano-SiC Coatings. *Surf. Coat. Technol.* **2012**, *213*, 33–40. [\[CrossRef\]](#)

42. García-Lecina, E.; García-Urrutia, I.; Díez, J.A.; Morgiel, J.; Indyka, P. A Comparative Study of the Effect of Mechanical and Ultrasound Agitation on the Properties of Electrodeposited Ni/Al₂O₃ Nanocomposite Coatings. *Surf. Coat. Technol.* **2012**, *206*, 2998–3005. [[CrossRef](#)]
43. Sajjadnejad, M.; Mozafari, A.; Omidvar, H.; Javanbakht, M. Preparation and Corrosion Resistance of Pulse Electrodeposited Zn and Zn–SiC Nanocomposite Coatings. *Appl. Surf. Sci.* **2014**, *300*, 1–7. [[CrossRef](#)]
44. Bezazi, A.; Boumediri, H.; Garcia del Pino, G.; Bezzazi, B.; Scarpa, F.; Reis, P.N.B.; Dufresne, A. Alkali Treatment Effect on Physicochemical and Tensile Properties of Date Palm Rachis Fibers. *J. Nat. Fibers* **2022**, *19*, 3770–3787. [[CrossRef](#)]
45. Bouhemame, N.; Aiadi, K.E.; Bezazi, A.; Boumediri, H.; Reis, P.N.B.; Imad, A.; Scarpa, F. Tensile Properties Optimization of Date Palm Leaflets Using Taguchi Method. *J. Nat. Fibers* **2022**, *19*, 6348–6364. [[CrossRef](#)]
46. Dehestani, M.; Khayati, G.R.; Sharafi, S. An Improved Optimization Model to Predict the Microhardness of Ni/Al₂O₃ Nanocomposite Coatings Prepared by Electrodeposition: A Hybrid Artificial Neural Network-Modified Particle Swarm Optimization Approach. *Measurement* **2021**, *179*, 109423. [[CrossRef](#)]
47. Rezgui, I.; Mihi, A.; Belloufi, A.; Abdelkrim, M.; Brabie, G. Fuzzy Logic Approach for Predicting the Effect of Manufacturing Parameters of Ni–Al₂O₃ Composite Coatings by Electrodeposition. *Int. J. Mod. Manuf. Technol.* **2017**, *9*, 65–72.

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.