Vol. 03, Iss. 4 (2025) 438-447, DOI: 10.61552/JME.2025.04.008

Journal of Materials and Engineering

www.jme.aspur.rs

Research article

Optimization of Tribo-Mechanical Behaviours of ENB Coatings Using GRA and ANOVA

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Keywords:

Electroless
Ni-B
Bath Parameter
Nano-indentation
COF
Grey Relational Analysis (GRA)
ANOVA

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Received: 2 September 2024
Revised: 6 October 2024
Accepted: 30 November 2024

A B S T R A C T

Electroless Ni-B (ENB) coatings are prepared over AISI 1040 steel having different compositions to estimate the impact of process parameters on the tribo-mechanical behaviours of coatings and to obtain a suitable set of bath parameters for optimized results. The surface hardness and coefficient of friction (COF) are selected as response parameters. The selected control parameters are NiCl2, NaBH4 and temperature. The control parameters are altered at 3 levels. The current study counts 3 control parameters having 3 levels of each parameter and the experimental combination was determined as per Taguchi's L9 orthogonal array (OA). Grey relational analysis (GRA) is employed to obtain the optimized values of control parameters to achieve a suitable set of response parameters. The GRA result reveals that the NaBH4 is the most significant bath parameter to control the coating behaviour followed by NiCl2 and coating bath temperature. The same result has been validated through ANOVA analysis.

1. INTRODUCTION

The coated layers are deposited to modify or improve the surface behaviours of any material. The coatings are applied to defend the base metal from corrosion, wear, alter friction behaviour, improve mechanical behaviours or scratch behaviour etc. The coatings in general may be soft or hard [1]. Normally, soft coatings are applied to provide soft-touch feelings like consumer electronics, mobile phone casing, domestic appliances, automotive interiors, packaging purposes etc. while the hard coatings may be used to improve anti-friction, wear resistance, surface behaviour enhancement purposes [1]. These surface behaviours may be achieved by providing a coated layer over the substrate through various methods and they are classified according to deposition techniques. The various coating methods are electroplating, electroless plating, PVD, CVD, solid lubricant coatings etc [1].

The formation of coating layers ove8r a substrate using a chemical reaction is known as electroless coating due to the absence of electricity [1-3]. The electroless coating method can be employed to deposit metals, alloys or composites over a large number of conductive as well as non-conductive specimens [1-4]. Electroless coatings like pure Nickel, Nickel-Boron, Nickel-Phosphorous are popular for its tribological, mechanical and thermal behaviours. The Electroless Ni-B (ENB) coated layers are largely used for its tribological applications [1, 3]. The existence of boron in the coating also improves its surface hardness which further improves its wear-resistant capability [1-3]. The ENB coatings may be deposited with $NaBH_4$ or Dimethylaminborane (DMAB) which acts as reducing agent to extract metal ions [1-4]. The boron concentration on coatings was observed to depend on reducing agent types [2-4]. The coating deposition rate is lower for DMAB reduced coatings than the borohydride reduced one [4]. Moreover, the DMAB reduced coating method uses an acidic bath and is deposited at a lower coating bath temperature of about 45ºC [5]. The borohydride reduced coating method uses an alkaline coating bath and comparatively at a higher temperature above 70ºC [6]. Usually, the amount of boron found in borohydride reduced Ni-B coatings is more compared to DMAB reduced coatings [4]. The coating deposition rate was also seen to alter due to chemical reaction with the variation in reducing agent content in the chemical solution [4, 7]. The chemical composition of the coating also varies with the reducing agent [4, 7]. The borohydride reduced electroless coating normally exhibit cauliflower-like surface morphology and columnar growth may also be observed [3, 4, 7, 8]. The columnar structure and cauliflower-like surface morphology are well known for good tribological behaviour due to reduced contact area [3, 4, 7, 8]. The ENB coatings are usually familiar for better surface hardness compared to mild steel [5, 7]. The same is found to improve further as $NaBH_4$ concentration increased which may be compared with hard chromium coatings [9]. The mechanical, as well as tribological properties, may change with the change in surface morphology, phase structure, grain size and surface texture [7, 8-10]. The same is observed to change with coating bath element concentration as well [7, 8, 10, 11]. The corrosion resistance of as-deposited ENB coatings is also found to alter due to change in phase, surface texture and amount of B in the coatings. The coating with higher B content possesses better corrosion resistance [3, 10, 11]. It is also seen that the amorphous phase of coated layers improves the corrosion behaviour compared to the coatings with crystalline structure [10, 11]. The surface roughness of coating also plays an important role to decide its corrosion behaviour. The surface with rough texture usually allows a corrosive medium to penetrate through surface cracks leading to lower corrosion resistance [3, 10, 11]. Hence, it can be seen that the behaviours of coatings deposited through the chemical process are dependent on chemical elements, surface texture, surface morphology, phase structure etc. [7, 11, 12]. The combination of chemical elements of ENB coatings may vary depending upon coating bath constitution as well as operating conditions [7, 11, 12]. The change in the chemical compounds of the coated films will result in a transformation of surface morphology, phase structure and ultimately led to a modification in its behaviours [7, 11, 12]. The surface hardness, and elastic modulus, may also be modified with the addition of hard nano-particles like ZrO_2, Al_2O_3, TiO_2 etc [13-15]. Therefore, the literature survey as mentioned above suggests that the coating behaviours are dependent mainly on coating bath composition. A small variation in coating bath composition has the capability to alter its surface structure and internal structure. This change may further lead to a change in coating behaviours. The study reveals that very little research has been carried out to determine a suitable set coating bath parameter concentration to achieve better coating characteristics. The ENB coating compositions have been optimized for tribological behaviours, surface hardness through Taguchi based GRA [16]. The coating behaviours are also optimized using fractal characterisation [17]. The GRA has been successfully and effectively employed to determine a suitable set of parameters for surface roughness in the EDM machining, turning operation or welding process [18, 19]. Taguchi based grey relational analysis, regression analysis and response surface methodology have been used for single or multi-response optimization purposes [18-21].

Hence, it may be observed clearly that the coating behaviour depend largely on bath composition which decides the chemical composition of the coated layers, morphology, phase structure etc. Therefore, it is important to determine a suitable composition to achieve desired coating behaviours. The optimization of coating behaviours is important to obtain desired coating characteristics. An attempt has been made in this current study to determine a suitable set of coating bath parameters that will provide the best-desired coating behaviours. Three process parameters viz. nickel chloride concentration, NaBH4 concentration and coating bath temperatures are varied with 3 levels each. The experiments were carried out following Taguchi's L9 OA. Then, GRA was used to evaluate the best process parameter values and their significance to control the response parameters viz. surface hardness and coefficient of friction. The obtained result has been further confirmed and validated through analysis of variance (ANOVA) analysis.

2. EXPERIMENTAL DETAILS

The current study contains the ENB coating film formation over steel substrate using the chemical deposition method. The mild steel substrates of dimensions of 15 x 15 x 2 mm^3 are utilised for mechanical test and cylindrical substrates of ϕ6 mm x 30 mm are utilised in friction and wear applications. The specimens are initially polished with fine grade emery papers for a smoother surface. The specimens are then washed using deionized water. The specimens were further processed with 50% HCl solution to get rid of any surface contamination and washed again. The substrates are emersed into moderately warm palladium chloride prior to the emersion of specimens into chemical solution. The elaborated coating development method is presented in previous studies [11]. The coating bath contains nickel chlorides ($NiCl_2$) which supply the nickel ion while the boron is supplied through the sodium borohydride ($NaBH_4$). Ethylenediamine ($C_2H_8N_2$) was used into the coating bath as a complexing agent. The coating bath contains sodium hydroxide (NaOH) as buffer. The addition of lead ions through lead nitrate at very little concentration prevents unexpected decomposition of bath. The coating bath temperature is monitored using an electric heater. The coatings were deposited for 4 hours for obtaining higher coating thickness and the coating bath was substituted with a new bath after 2 hours.

Table 1. Process parameters with levels.

Parameters	Units	Coded Name	Low Level	Mid-Level	High Level
Nickel Chloride	g/l	A	10	20	30
Sodium Borohydride	g/l	B	0.40	0.80	1.20
Bath Temperature	°C	C	75	85	95

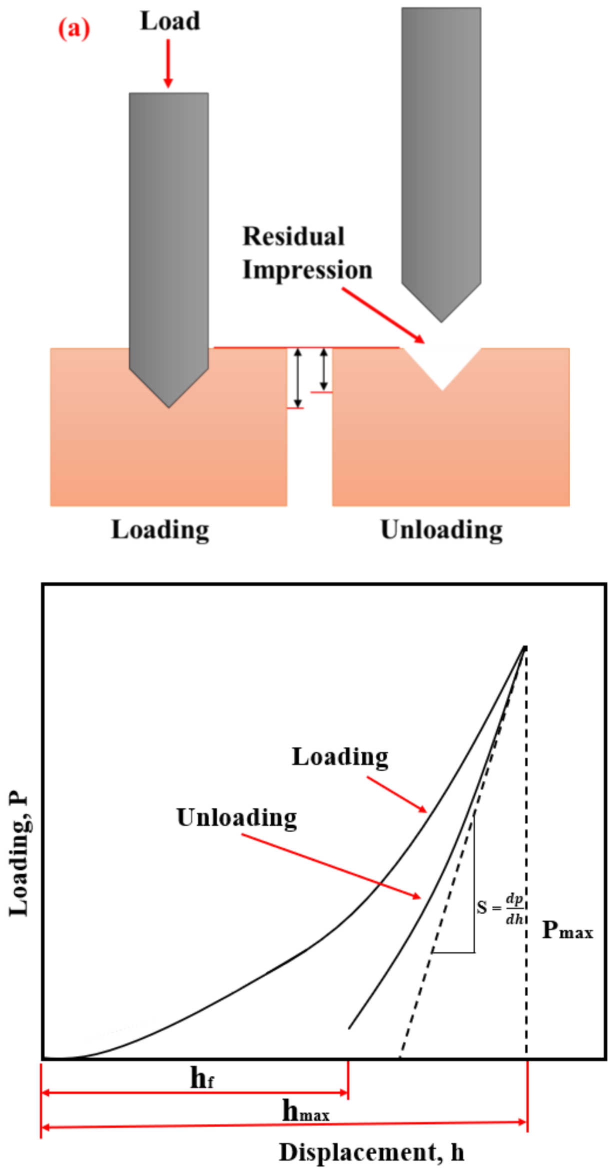

Fig. 1. (a) Working Principle and (b) Loading unloading curve.

Three different coating bath parameters (nickel chloride, sodium borohydride, coating bath temperature) were selected as control parameters. The response variables considered are nano-hardness (Hv) and coefficient of friction (COF). The control parameters are displayed in Table 1. In the present study, Taguchi L9 orthogonal array has been selected for conducting experiments that requires 9 sets of experiments. The surface hardness tests are conducted in a nano-indentation tester. The experimental set-up uses Berkovich indenter for the indentation purpose. The working of nano-indentation technique is displayed in Figure 1(a). The loading unloading curve displayed in Figure 1(b) is obtained from nano-indentation test and it is applied for the determination of surface hardness of the coatings. Each test is carried out as per indentation depth-based method with a loading unloading rate of 20 mN/min. The indentation depth is considered to be 500 nm to maintain the indentation depth of lower than 1/10th of least coating thickness [22] as it eliminates the chances of substrate effect.

Fig. 2. Multi-tribo Tester.

Tribological test of the coated substrates are performed in a pin-on-disc type multi-tribotester displayed in Figure 2 and the test results are recorded in a dedicated computer attached with the machine. The test is carried out against an applied load of 50 N, sliding speed of 39.25 cm/s. The test is conducted following ASTM G99-05 (Reapproved 2010).

All set of tests are carried out for at least 3 times and the average value of the nano-hardness and coefficient of friction (COF) are presented in this manuscript. The test results are displayed in Table 2. These results are utilised to calculate an optimum set of bath parameters and significance of bath parameters through GRA and ANOVA. The characterisation is carried out for the specimens obtained at low level values (A -10 g/l, B - 0.4 g/l and C- 75ºC), mid-level values (A -20 g/l, B - 0.8 g/l and C- 85ºC) and high-level values (A -30 g/l, B - 1.2 g/l and C- 95ºC) of coating bath parameters only. Those coatings are then characterised for elemental analysis, surface morphology, as well as phase structures. The minimum coated layer thickness is also determined with SEM.

Table 2. Experimental Combinations and responses.

Sl. No	A	B	C	Hv	COF	
1	10	0.4	75	571.9	0.513	Low Level
2	10	0.8	85	920.59	0.784	
3	10	1.2	95	1181	0.625	
4	20	0.4	85	659.66	0.655	
5	20	0.8	95	872.87	0.818	
6	20	1.2	75	1206.9	0.632	
7	30	0.4	95	753.49	0.629	
8	30	0.8	75	928.45	0.763	
9	30	1.2	85	1251.7	0.539	
10	20	0.8	85	784.13	0.663	Mid-Level

3. OPTIMIZATION METHODOLOGY

The recorded experimental results of three different input parameters were utilised to determine an optimum set of process parameters. The optimization was conducted through GRA which is a technique to deal with uncertain as well as multi-response systems efficiently and effectively. The technique was introduced by Deng Julong in 1989 [23]. The GRA system deals with a system having partial information that lies between black and white i.e. grey. Black indicates no information and white indicates full information. The correlation amongst the coating deposition control parameters as well as the coating behaviours are complicated which can be modelled efficiently and effectively by GRA [16, 24-26]. The multi-response optimization is conducted through GRA. The results of different units and different ranges are normalized and converted to a single index i.e. grey relational grade (GRG) which is utilised for further analysis.

The GRA method involves four steps. The 1st step involves the normalization of experimental results having different data ranges and units. The normalization of response parameters is done following 3 different criteria namely higher-the-better, lower-the-better and nominal-the-better. The quality characteristics are selected based on the desired response parameter [25, 26]. In this current study, the nano-hardness values are normalized using the higher-the-better criterion as the higher values of nano-hardness are desired. The coefficient of friction (COF) values is normalised using the lower-the-better criterion as the lower values of COF are desired.

The higher-the-better criterion is presented in Equation 1:

$$x_i^*(k) = \frac{x_i^o(k) - Min x_i^o(k)}{Max x_i^o(k) - Min\ x_i^o(k)} \quad (1)$$

where $x_i^*(k)$ normalized value, $Min x_i^o(k)$ and $Max x_i^o(k)$ are the smallest and highest value of the response.

The lower-the-better criterion is presented in Equation 2 and given as

$$x_i^*(k) = \frac{Max x_i^o(k) - x_i^o(k)}{Max x_i^o(k) - Min\ x_i^o(k)} \quad (2)$$

The normalized values of all the responses are presented in Table 3. The normalized value of the responses is further utilised to calculate the grey relational coefficients (GRC) using the following Equation 3:

$$\xi_i(k) = \frac{\Delta_{min}(k) + \zeta.\Delta_{max}}{\Delta_{oi}(k) + \zeta.\Delta_{max}} \quad (3)$$

where $\xi_i(k)$ = grey relational coefficient value, Δ_{oi}(k) = difference of absolute $x_i^*(k)$ and $x_o(k)$, Δ_{min} and Δ_{max} are the lowest and greatest value of the absolute difference (Δ_{oi}). In the equation (ii), ζ = distinguishing coefficient where ζ € {0, 1} [25, 26] which controls the effect of Δ_{max}. The value of ζ is taken to be 0.5 for this present study [25, 26]. The calculated values of GRA are presented in Table 3. The higher value of grey relational grade (GRG) indicates the nearer optimum condition.

4. RESULTS AND DISCUSSIONS

The coated layer thickness obtained at low level is presented in Figure 3 which shows that the coating obtained at low-level concentration have a coating thickness of 6.96 μm. The coating thickness is calculated from mass gains and cross checked through SEM. The coating thickness is seen to rise with coating bath level. The improvement in thickness is an indication of an improvement in reduction rate leading to an improvement in plating rate with the raised bath level as the deposition time is kept constant at 4 hours [3, 8, 11]. The $NaBH_4$ acts as a reducing agent and it increases the reduction rate at higher borohydride content which leads to a rise in coating deposition rate [3, 7, 8, 11].

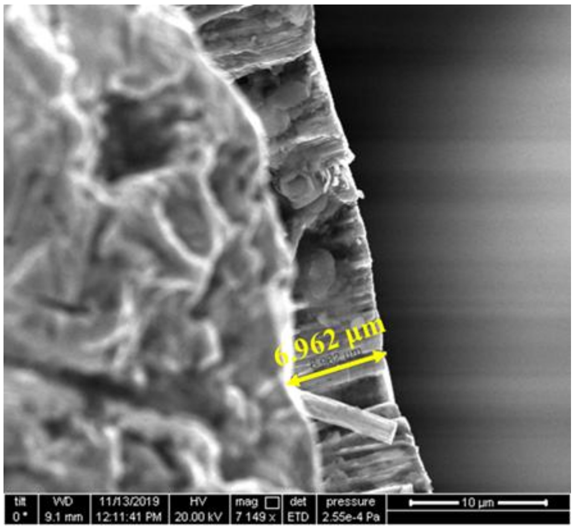

Fig. 3. Cross-Cut Coating Thickness.

The coated surface morphology observed under SEM is displayed in Figure 4, which represents that the coatings are uniform as well as homogeneously distributed throughout their surface [3, 7, 8, 11]. The coatings at all three levels exhibit cauliflower-like surface morphology [3, 7, 8, 11]. Borohydride reduced electroless coatings generally possess cauliflower-like morphology and they are known for reducing the COF value due to a decrease in actual surface contact area [7, 27, 28]. The cauliflower-like surface morphology also makes the coated surface to be self-lubricious which also reduces the friction coefficient. It can also be seen in Figure 4 that the nodules of the coated surfaces increased in size with the coating bath level. The higher size nodules at high-level concentration forms granular structure which reduces the contact area and reduces the friction further [7, 27, 28].

The elemental analysis is carried out through EDAX which confirms the existence of nickel, boron, carbon and oxygen in the as-deposited coatings [7, 11, 29]. EDAX analysis results at 3 bath levels are displayed in Figure 5. The coatings possess 3.60% to 3.90%, 5.60% to 5.90% and 6.90% to 7.30% of boron at low, medium and high level, respectively. Ni content is found to be 91.40% to 94.50%, 88.90% to 92.70% and 87.60% to 89.40% at low, medium and high concentration levels, respectively. The coatings also contain a very negligible amount of carbon and oxygen which might have come from some external source. The elemental analysis result shows an increase in boron content with increased $NaBH_4$ [7, 11]. This rise of boron concentration in the coatings may lead to an improvement in the hardness which may further improve the wear resistance of coated specimens.

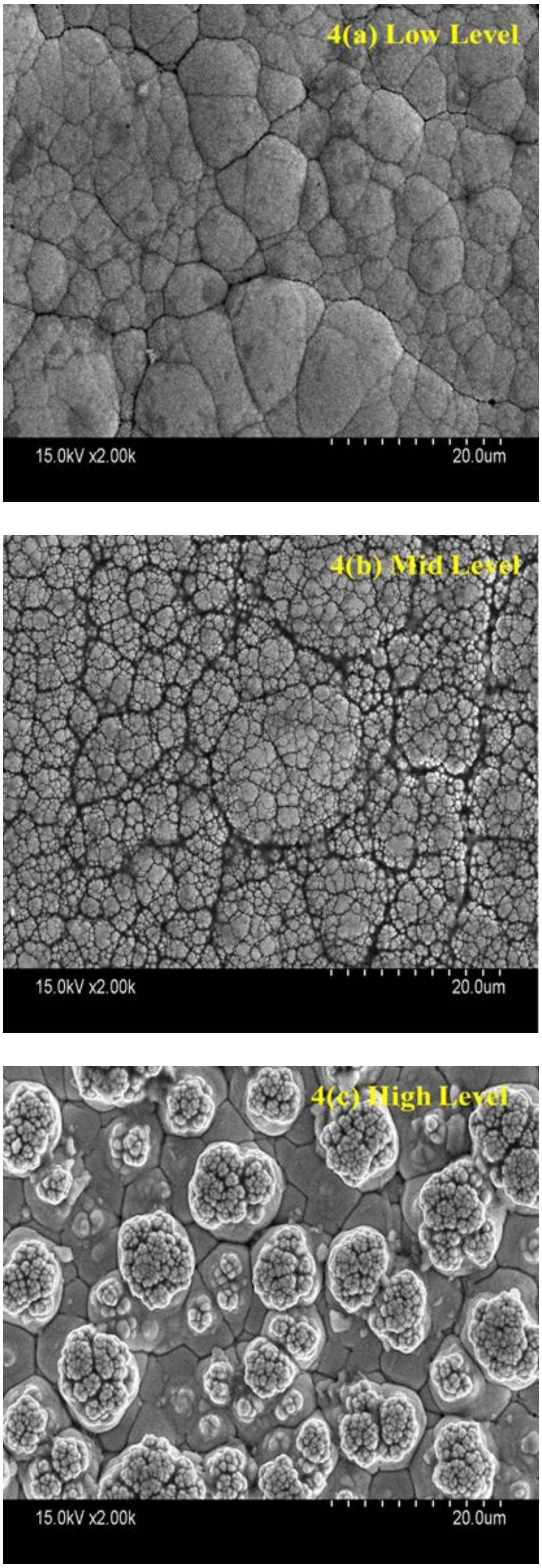

Fig. 4. SEM Image of Surface Morphology.

The XRD plots of the coated substrates are presented in Figure 6. The XRD plot exhibit a broad hump with a high-intensity crystalline peak for the coatings obtained with low $NaBH_4$ concentrations. This type of XRD pattern indicates the coexistence of amorphous and nano-crystalline phase structures. This trend agrees well with the previous study [7, 11, 28, 30].

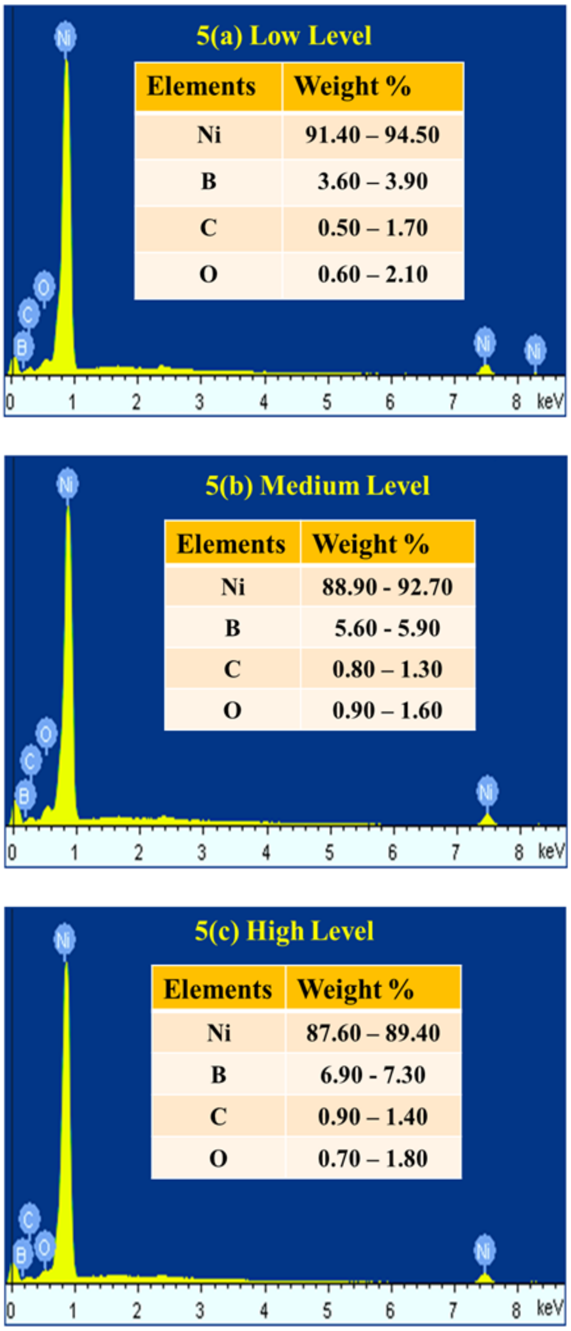

Fig. 5. EDAX Spectrum.

The coatings having a boron content of more than 4 wt.% normally possess an amorphous structure [30, 31]. The broad peak diminished gradually with increased $NaBH_4$ content. The decrease in crystalline peak with $NaBH_4$ concentration indicates the modification in phase structure from crystalline to amorphous. This transformation of phase may be correlated with the rise in boron.

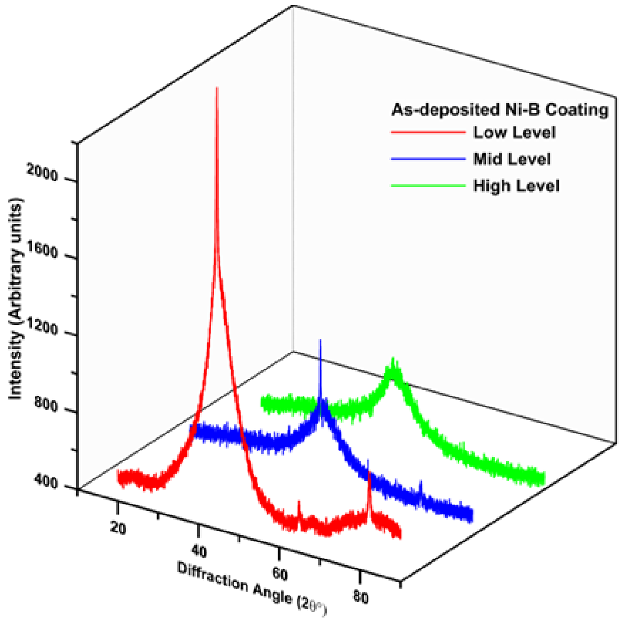

Fig. 6. XRD Plot.

The hardness of the coatings is determined through a nano-hardness tester with the highest indentation depth of 500 nm and the data obtained using the loading-unloading curve. The loading-unloading curve for coating at different levels is displayed in Figure 7. The loading-unloading curve also represents the maximum applied load necessary for the coated specimens at the highest coating bath concentration level for the same indentation depth and it reduced with bath level. This confirms the enhancement in surface hardness of the coatings due to rise in bath level resulting a rise in boron content in as-deposited coatings.

The effect of each process parameter may be determined using the orthogonal array at different levels. The obtained experimental results are utilised to calculate a normalised value of different response parameters. The normalised value is further utilised to calculate the GRCs and GRGs for different set of experiments. The calculated values are presented in Table 3.

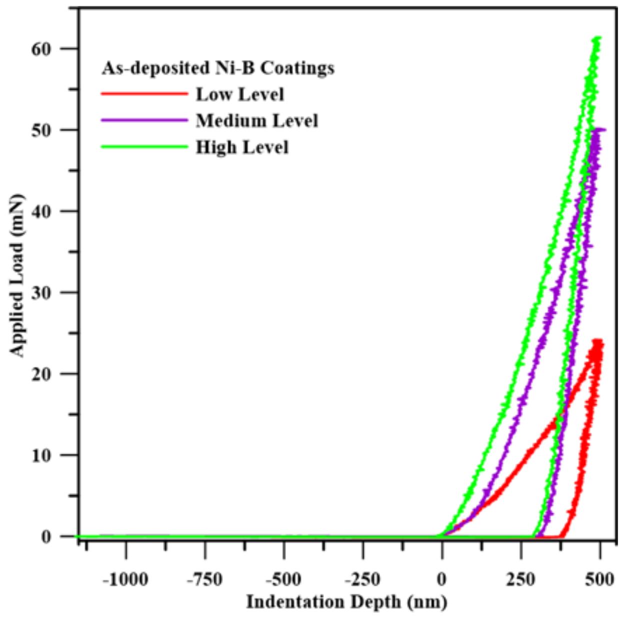

Fig. 7. Loading Unloading Curve

The mean of GRG values is to be calculated for different level of bath parameters for the estimation of the importance and contribution of those parameters. The mean GRG of nickel chloride (A) at low level is determined by calculating the mean of the first 3 experiments. Likewise, the mean GRG of all the process parameters is calculated which are presented in Table 4. The delta value of the mean GRG can also be calculated from the difference between the largest to smallest of each column. The delta value was utilised for calculating the rank of the process parameters. The mean of GRG values is presented in Table 4. The higher value of delta indicates the higher significance of control parameters on response variables [32].

Table 3. Grey Relational Generation and Grades.

Sl. No.	Normalized Values		GRC		GRG	Rank
	Hv	COF	Hv	COF		
1	0.000	1.000	1.000	0.333	0.667	2
2	0.513	0.111	0.494	0.818	0.656	3
3	0.896	0.633	0.358	0.441	0.400	8
4	0.129	0.534	0.795	0.483	0.639	4
5	0.443	0.000	0.530	1.000	0.765	1
6	0.934	0.610	0.349	0.451	0.400	7
7	0.267	0.620	0.652	0.447	0.549	6
8	0.524	0.180	0.488	0.735	0.611	5
9	1.000	0.915	0.333	0.353	0.343	9

The sodium borohydride was found to have the highest delta value which confirms that sodium borohydride is the most important parameter to decide the response variables while $NiCl_2$ was observed to be the second most significant parameter followed by coating bath temperature. The mean effect plot is also presented in Figure 8. The optimized set of parameters for the best set of results for response variables are found to be $A_2B_2C_3$ ($NiCl_2$ = 20 g/l, $NaBH_4$ = 0.80 g/l and coating bath temperature = 95ºC). The same corresponds to experiment no 5 in the current L9 experimental design matrix. The slope or curves in the mean effect plot also indicates the significance of process parameters [32]. Higher slope indicates the higher significance of parameters. Hence, the highest significance of sodium borohydride can also be observed from Figure 8 and the same trend can be seen from Table 4 as well.

Table 4. Mean GRG Values.

Level	A	B	C
1	0.574	0.618	0.559
2	0.601	0.677	0.546
3	0.501	0.571	0.571
Delta	0.100	0.106	0.025
Rank	2	1	3

The GRA result shows that sodium borohydride is the most significant parameter to control the tribo-mechanical behaviour of coated layers. Therefore, it may be said that the increase in nano-hardness and reduction in COF value, were mainly because of the variation in $NaBH_4$ concentration. The best set of parameters obtained from GRA had the highest concentration of sodium borohydride which led to the best set of mechanical properties of coatings.

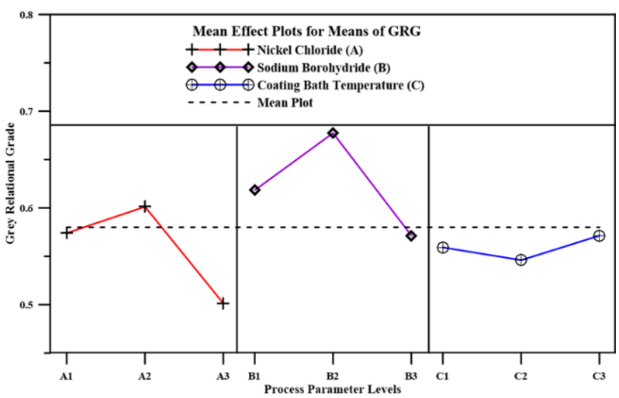

Fig. 8. Mean effect plot for Means of GRGs.

The importance of the coating bath parameters is estimated using ANOVA. ANOVA analysis is conducted using the obtained GRG value from GRA. The results of ANOVA using GRG are displayed in Table 5. ANOVA result usually contains the degrees of freedom (DOF) of the control parameters, sum of squares (SS), mean squares (MS), F-ratio and contribution. The F-ratio indicates the variance ratio or the ratio of mean square and the mean square error [21, 24-26, 32]. For a specific confidence level, F-ratio indicates the importance of control parameters i.e. higher value of F-ratio indicates higher level of significance. In the current investigation, sodium borohydride is found to be the most influential parameter with a confidence level of 95% and with 84.95% contribution. The nickel chloride and coating bath temperature contributes only 9.20% and 5.54% to the nano-hardness and COF value of coatings.

Table 5. ANOVA Results.

Parameters	DOF	SS	MS	F-ratio	P	Contribution (%)
A	2	0.016013	0.008006	3.07	0.245	9.20
B	2	0.147784	0.073892	28.37	0.038	84.95
C	2	0.009640	0.0005821	0.22	0.784	5.54
Error	2	0.000520	0.002605			
Total	8	0.173957				

The final step for optimization through GRA is the confirmation test which is conducted to estimate whether there is any enhancement in result. Generally, mid-level concentrations are chosen as the initial test condition. The GRG of the optimized set of parameters may be calculated using the following Equation 4 given as:

$$\acute{\eta} = \eta_m + \sum_{i=1}^{0}(\eta_i - \eta_m) \tag{4}$$

where ηm is the mean grade of GRGs, ηi is the mean grade at the optimal level of all individual control parameters, and 0 is the number of main design parameters that significantly affect the coating behaviours.

Table 6. Confirmation Test Results.

	Initial Level	Optimal Parametric Level	
		Predicted	Experimental
Level	A2B2C2	A2B2C3	A2B2C3
Hv	784.13		
COF	0.663		
Grade	0.556	0.689	0.765

Improvement of grey relational grade = 37.59%

The confirmation test results for the current study are displayed in Table 6. The predicted as well as the experimental grade is in well agreement with each other. A very high improvement in the GRG of 37.59% is obtained which indicates the significance and applicability of the GRA in successfully optimizing the coating process parameters.

5. CONCLUSIONS

In this current study, the coating parameters were successfully and efficiently optimized through GRA. The control parameters selected are $NiCl_2$, $NaBH_4$ and coating bath temperature. The coatings are deposited over steel substrate and tested for nano-hardness and COF values. The process parameters are optimized for higher values of the response variables. The optimized set of control parameters are $A_2B_2C_3$ i.e. $NiCl_2$ = 20 g/l, $NaBH_4$ = 0.80 g/l and coating bath temperature = 95ºC. Moreover, sodium borohydride was observed to be the most influential parameter to control the response parameters followed by $NiCl_2$ and coating bath temperature. The amount of $NaBH_4$ present in the coating bath solution acted as main constituent to alter tribo-mechanical behaviour of coating with a contribution of 84.95% followed by $NiCl_2$ and coating bath temperature with 9.20% and 5.54%, respectively. The overall improvement of 37.59% may also be observed compared to initial test conditions.

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