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OPTIMIZATION OF ELECTROLESS Ni-B COATINGS BASED ON MUTIPLE ROUGHNESS CHARACTERISTICS

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ABSTRACT

This paper presents an experimental study of multiple roughness characteristics of electroless Ni–B coatings. Optimization of coating process parameters based on the Taguchi method combined with grey relational analysis is done to minimize roughness. Experiments are carried out by utilizing the combination of process parameters based on the L_{27} Taguchi orthogonal array design with three process parameters, namely, bath temperature, concentration of reducing agent and concentration of nickel source. It is observed that concentration of reducing agent has the most significant influence in controlling roughness characteristics of electroless Ni-B coating. The optimum combination of process parameters for minimum roughness is obtained from the analysis. The surface morphology and phase structure of coatings are also studied with the help of scanning electron microscopy and x-ray diffraction analysis.

Keywords: Electroless Coating, Ni-B, Roughness, Taguchi Method, Grey Analysis.

1. INTRODUCTION

The invention of Electroless Nickel (EN) coating is mainly credited to Brenner and Riddell [1]. Borohydride reduced coatings or Ni-B coatings have found extensive applications in aerospace, automotive, chemical and electrical industries especially due to their high hardness, wear resistance, solderability, abrasion resistance, etc.[2-10].

Surface roughness has a large impact on the mechanical properties such as fatigue behaviour, corrosion resistance, creep life, etc. It also affects other functional attributes of machine components such as friction, wear, light reflection, heat transmission, lubrication, electrical conductivity, etc. As a result, there is a need for research on modeling surface roughness and optimization of the controlling parameters to obtain a surface finish of the desired level. Hence the present study is directed towards roughness optimization of Ni-B coatings.

The present study uses Taguchi method to optimize the process parameters in order to minimize the surface roughness of Ni-B coating. Taguchi method [11] is a powerful tool for design of high quality systems based on orthogonal array (OA). Taguchi makes use of S/N (Signal to Noise) ratio to denote the quality characteristics. The S/N ratio characteristics can be divided into three categories: lower-the-better (LB), higher-the better (HB) and nominal-the best (NB). Grey relational analysis is done to have an optimum coating parameter combination that yields optimum roughness characteristics. A statistical analysis of variance (ANOVA) [12] is performed to find which process parameters are statistically significant. Finally, a confirmation experiment is conducted to verify the optimal process parameters obtained from the parameter design.

2. EXPERIMENTAL DETAILS

2.1 Coating Deposition

Mild Steel (AISI 1040) is used as the substrate material (square blocks of size 20 mm × 20 mm × 8 mm) for the deposition of Ni-B films. The samples are cleaned of any foreign particles and corrosion products prior to coating. Table 1 indicates the bath composition and the operating conditions for successful coating of electroless Ni-B. The cleaned samples were activated in palladium chloride solution and placed in the bath for a deposition time of 1 hour. For each sample, the procedure is repeated twice except for the palladium chloride activation in the second round. The range of coating thickness was found to lie between 20-25 microns. After the deposition the samples were taken out of the bath and cleaned using distilled water. To see the effect of heat treatment on the characteristics of the coating, some of the samples were heat treated at 450°C (for 1 hour) in a box furnace.

It is important to note that the present study does not consider substrate roughness as the input variable. Hence it is required that all samples have uniform roughness. This is achieved by preparing large number of samples and selecting those which show insignificant variation (less than 0.1%) in roughness.

2.2 Design Parameters

Although many factors can affect the quality of EN coating, the present study considers (A) bath temperature

(B) reducing agent concentration and (C) nickel source concentration as the process parameters based on an intensive literature review. These three factors were chosen as the main design factors along with their interactions in the present study. Table 2 shows the design factors along with their levels.

Table 1: Bath constituents and ranges of values for electroless Ni-B coating

Parameters	Ranges of parameters
Nickel chloride	15 – 25 g/l
Sodium	0.6 – 1.0 g/l
Ethylenediamine	59 g/l
Lead nitrate	0.0145 g/l
Sodium hydroxide	40 g/l
Bath temperature	85 – 95 °C
pH of solution	12.5
Deposition time	1 hr

Table 2: Design factors and their levels

Design Factors	Unit	Levels		
		1	2	3
Bath Temperature (A)	ºC	85	90^a	95
Reducer conc. (B)	(g/l)	0.6	0.8^a	1.0
Nickel source conc. (C)	(g/l)	15	20^a	25

a : initial condition

2.3 Response Variable

The present study is carried out to consider the roughness characteristics of electroless Ni-B coatings as the performance characteristics. Since a single parameter can't totally describe the quality of a surface, five roughness parameters are considered. It is a multiresponse problem and the five roughness parameters considered are: Centre line average roughness (R_a), Root mean square roughness (R_q), Skewness (R_{sk}), Kurtosis (R_{ku}) and Mean line peak spacing (R_{sm}).

2.4 Design of Experiment

Taguchi method uses a set of orthogonal arrays (OA) to reduce the number of experiments required which may otherwise increase the time and cost of the experiment. An OA provides the shortest possible matrix of combinations in which all the parameters are varied to consider their direct effect as well as interactions simultaneously. Taguchi has tabulated several standard OAs. In this study, a L_{27} OA, which has 27 rows corresponding to the number of tests and 26 degrees of freedom (DOFs) with 13 columns at three levels, is chosen. As per the requirements of the L_{27} OA, the 1st column is assigned to bath temperature (A), the 2nd column is assigned to concentration of reducing agent (sodium borohydride) (B), the 5th column is assigned to concentration of source of nickel (nickel chloride) (C) while the rest of the columns are assigned to the two-way interactions of the factors and error terms.

2.5 Roughness Measurement

Roughness measurement is done using a portable stylus and skid type profilometer, Talysurf (Taylor Hobson, Surtronic 3+). The profilometer is set to a cut-off length of 0.8 mm, Gaussian filter, and traverse speed 1 mm/s with 4mm evaluation length. Roughness measurement on the electroless coatings is repeated four times and the average of four measurements is recorded. The measured profile is digitized and processed through the dedicated advanced surface finish analysis software Talyprofile for evaluation of the roughness parameters.

2.6 Surface Morphology and Compound Analysis

Study of surface morphology of the EN coatings is obtained by SEM (JEOL, JSM-6360) to analyse the microstructure of the deposited coatings both before and after annealing at 450ºC, in order to see the effect of annealing temperature. The different precipitated phases of the deposits both before and after annealing were analyzed by X-ray diffraction (XRD) analyzer (Rigaku, Ultima III).

3. RESULTS AND DISCUSSION

3.1 Grey Relational Analysis

The objective of the present study is to minimize the five roughness parameters (R_a, R_q, R_{sk}, R_{ku}, R_{sm}) simultaneously by optimizing the coating process parameters. Hence it is a case of multiresponse optimization which cannot be handled by Taguchi method alone. Hence the Grey relational analysis [13] is used together with Taguchi method to solve the problem. The Grey theory has been proven to be useful for dealing with poor, incomplete and uncertain information. In Grey analysis black represents having no information and white represents having all information. A grey system has a level of information between black and white and hence the name "Grey" is associated with it. The main objective of grey system theory is to supply information so that one can whiten the greyness.

In grey relational analysis the first step is to perform the grey relational generation in which the results of the experiments are normalized in the range between zero and one. The second step is to calculate the grey relational coefficient from the normalized data to represent the correlation between the desired and actual experimental data. The overall grey relational grade is then computed by averaging the grey relational coefficient corresponding to each performance characteristic. Overall evaluation of the multiple performance characteristics is based on the calculated grey relational grade. As a result, optimization of the complicated multiple performance characteristics is converted into optimization of a single grey relational grade. The optimal level of the process parameters is the level with the highest grey relational grade. Furthermore, a statistical ANOVA is performed to find which process parameters are statistically significant. With the grey relational analysis and statistical ANOVA, the optimal combination of the process parameters can be predicted. Finally, a confirmation experiment is conducted to verify the optimal process parameters obtained from the analysis. For conciseness, the details of the Grey analysis and the intermediate calculations are omitted here. Only the final values for the grey relational grade and their order are listed in Table 3.

3.2 Analysis of Signal to Noise Ratio

Signal to noise ratio is preferred by Taguchi instead of mean since the former can capture the variability within a trial condition. In the present work S/N ratio analysis is done with grey relational grade as the performance index. The S/N ratio for grey relational grade is calculated using HB (Higher the better) criterion and is given by

$$S/N = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \qquad (1)$$

where y is the observed data and n is the number of observations. As the experimental design is orthogonal, it is possible to separate out the effect of each coating parameter at different levels. For example, the mean grey relational grade for factor A at levels 1, 2 and 3 can be calculated by averaging the grey relational grades for the experiments 1–9, 10–18 and 19–27, respectively. The mean grey relational grade for each level of the other factors can be computed in the similar manner. The grey relational grade for each level of the factors A, B, and C is summarized in Table 4. All the calculations are performed using Minitab® software [14]. The mean response table also includes ranks based on Delta statistics, which compare the relative magnitude of effects. The Delta statistic is the highest average for each factor minus the lowest average for the same. Ranks are assigned based on Delta values; rank 1 is assigned to the highest Delta value, rank 2 to the second highest Delta value, and so on. The corresponding main effects and interaction effects plots between the process parameters are also shown in Fig 1 and Fig 2 respectively. In the main effects plot if the line for particular parameter is near horizontal, then the parameter has no significant effect. On the other hand, a parameter for which the line has the highest inclination will have the most significant effect. From the main effects plot (Fig 1) it is clear that factor B i.e. the concentration of the reducing agent is the most significant factor while factor A i.e. bath temperature is also very significant. In case of interaction plots non-parallelism of the parameter effects are observed. If the lines on the interaction plots are non-parallel, interaction occurs and if the lines cross, strong interactions occur between parameters. From the interaction plots (Fig 2) it can be observed that the interaction between A and B are somewhat significant. Thus from the present analysis it is clear that the concentration of reducing agent (B) is the most influencing parameter for roughness parameters of electroless Ni-B coatings. The optimal process parameter combination for minimum roughness characteristics is found to be A2B1C2.

Table 3: Grey relational grade and its order

Exp. No.	Grey relational grade	Order
1	0.5365	21
2	0.7204	8
3	0.6224	16
4	0.5053	23
5	0.6739	13
6	0.4997	24
7	0.5270	22
8	0.4436	27
9	0.5767	19
10	0.7752	2
11	0.7326	6
12	0.7701	3
13	0.6179	17
14	0.6936	10
15	0.6740	12
16	0.5600	20
17	0.7570	5
18	0.6969	9
19	0.7572	4
20	0.6753	11
21	0.8553	1
22	0.6474	14
23	0.7314	7
24	0.6238	15
25	0.5937	18
26	0.4541	26
27	0.4545	25

Table 4: Response table for the grey relational grade

Level	A	B	C
1	0.5673	0.7162	0.6134
2	0.6975	0.6297	0.6536
3	0.6437	0.5627	0.6415
Delta	0.1302	0.1535	0.0402
Rank	2	1	3

Total mean grey relational grade = 0.6361

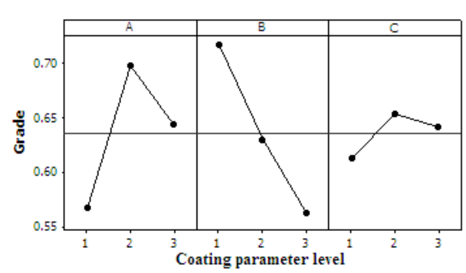

Fig 1. Main effects plot for mean grey relational grade

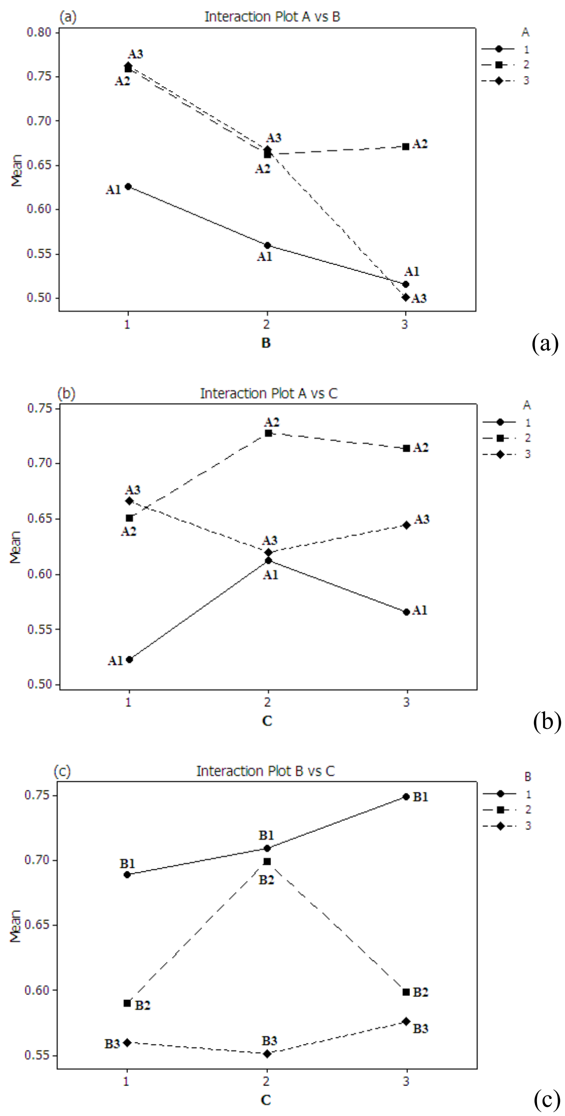

Fig 2. Interaction effects plot for mean grey relational grade (a) A versus B, (b) A versus C and (c) B versus C.

3.3 Analysis of Variance (ANOVA)

ANOVA is used to quantify the significance of the process parameters and their interactions, on the performance characteristics considered. This is accomplished by separating the total variability of the response which is measured by the sum of the squared deviations from the total mean of the response, into contributions by each of the process parameters and the error. In the present study ANOVA is performed using Minitab. ANOVA results for grey relational grade are shown in Table 5. In ANOVA a ratio called F-ratio, which is the ratio between the regression mean square and mean square error is used to measure the significance of the parameters under investigation with respect to the variance of all the terms included in the error term at the desired significance level, α. A calculated F-ratio which is higher than the tabulated F-ratio indicates that the factor is significant at desired α level. ANOVA table also shows the percentage contribution of each parameter. It is seen that parameter B, i.e. concentration of reducing agent has got the most significant influence on roughness at the confidence level of 95% within the specific test range. Also parameter A, i.e. bath temperature is significant at the same confidence level. None of the interaction is found to have significant effect on the roughness characteristics of electroless Ni-B coating but interaction between the bath temperature and the concentration of reducing agent (A × B) has got somewhat contribution in controlling the roughness parameters.

Table 5: Results of ANOVA for grey relational grade

Source	df	SS	MS	F	% contribution
A	2	0.077	0.038	5.18*	24
B	2	0.106	0.053	7.16*	33
C	2	0.008	0.004	0.51	2.5
A*B	4	0.035	0.018	1.17	11
A*C	4	0.018	0.004	0.59	5.5
B*C	4	0.021	0.005	0.70	6.5
Error	8	0.059	0.007		
Total	26	0.324			

* - significant at 95% confidence level ($F_{0.05,2,8} = 4.46$)

3.4 Confirmation Test

After the optimal level of the process parameters is selected, the final step is to predict and verify the improvement of the performance characteristic using the optimal level of the process parameters. The estimated grey relational grade $\hat{\gamma}$ using the optimal level of the process parameters can be calculated as:

$$\hat{\gamma} = \gamma_m + \sum_{i=1}^{o} \left(\bar{\gamma}_i - \gamma_m\right) \qquad (2)$$

where γ_m is the total mean grey relational grade, $\bar{\gamma}_i$ is the mean grey relational grade at the optimal level, and o is the number of the main design parameters that significantly affect the roughness characteristics of electroless Ni-B coating. Table 6 shows the comparison of the estimated grey relational grade with the actual grey relational grade using the optimal parameters. It can be observed that there is a good agreement between the estimated and the actual grey relational grade. The increase of the grey relational grade from the initial coating parameters to the optimal coating parameters is 0.04 which is about 6% of the mean grey relational grade.

3.5 Surface Morphology and Phase Content

Surface morphology study of the coatings is done by SEM in order to analyse the effect of heat treatment on the microstructure of the coatings for some of the samples at random and they show similar qualitative change in microstructure. Fig 3 shows the SEM micrographs of the samples in as deposited and under heat treated conditions. It is seen that the electroless Ni-B coatings in general exhibit a defect free surface with distribution of Ni-B nodules, more like that of a cauliflower surface which indicates that the coating possesses a lubricious behaviour. The surface of the coating appears dense and light grey in colour with low porosity. When heat treated, the Ni-B nodules grow in size giving rise to a coarse-grained structure. This indicates that in as deposited condition the structure is a mixture of amorphous and microcrystalline which becomes crystalline with heat treatment. This is further confirmed by the XRD patterns of Ni-B deposits in as deposited and heat treated condition (Fig 4). The XRD patterns in as deposited condition is a collection of microcrystalline peaks. But with heat treatment at 450°C for one hour, broad peaks of Ni, Ni_2B and Ni_3B are observed.

Table 6: Results of confirmation test

Level	Initial parameter combination	Optimal parameter	
		Prediction	Experimentation
	A2B2C2	A2B1C2	
R_a	0.420		0.405
R_q	0.526		0.522
R_{sk}	0.651		0.397
R_{ku}	6.100		3.620
R_{sm}	0.050		0.084
Grade	0.693	0.777	0.733

Improvement of grey relational grade = 0.04

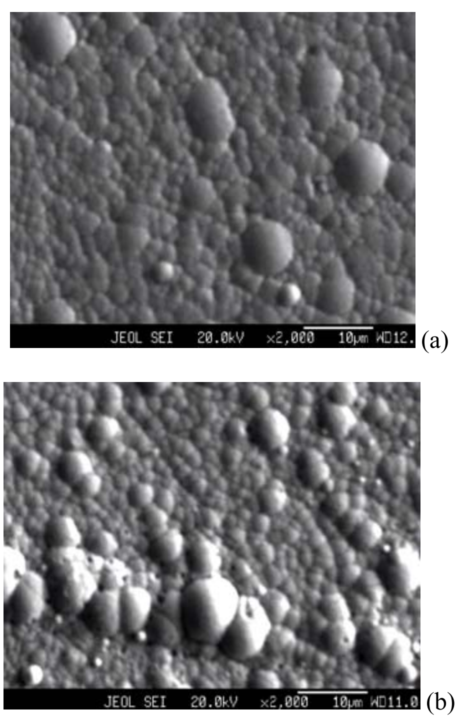

Fig 3. SEM micrographs of the coating surfaces: (a) as-deposited and (d) heat treated at 450ºC.

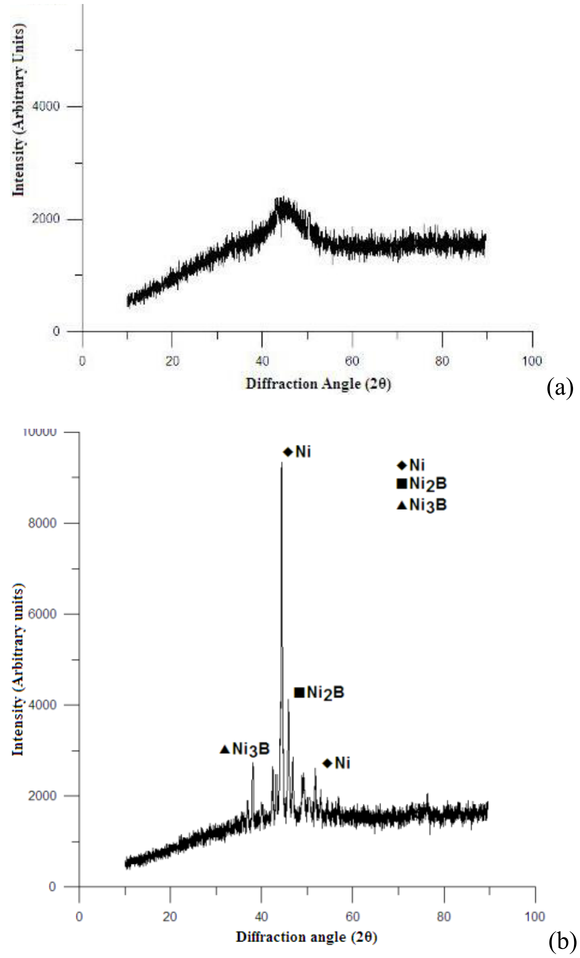

Fig 4. XRD patterns of electroless Ni–B deposit in (a) as-deposited and (b) heat treated at 450ºC

4. CONCLUSION

In the present study Taguchi orthogonal array together with grey relational analysis is used to optimize the coating process parameters (bath temperature, concentration of reducing agent and concentration of nickel source) together in order to optimize multiple roughness parameters (R_a, R_q, R_{sk}, R_{ku}, R_{sm}) of electroless Ni-B coating. The following conclusions are reported :

- Concentration of reducing agent (B) is the most important parameter that significantly affects the roughness characteristics at a confidence level of 95%. Also the bath temperature (A) is very significant at the same level of confidence.
- Interaction between bath temperature and concentration of reducing agent (A × B) has got somewhat contribution in controlling the roughness features.
- The optimal parameter combination for minimum friction is A2B1C2.
- The optimal parameter combination yields about 0.04 grey relational grade more than the initial condition.

The microstructural study through SEM micrographs reveals that the coating has a cauliflower like structure with no obvious surface damage and low porosity. Also the coating is dense and light grey in colour. In as deposited condition the coating is a mixture of amorphous and microcrystalline structure which generally turns crystalline with heat treatment. This is confirmed by the presence of Ni_2B and Ni_3B phases in the XRD plot of Ni-B coating annealed at 450°C.

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